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The Journal *of the* Society of Dyers and Colourists

Volume 74 Number 8

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THE SOCIETY OF DYERS AND COLOURISTS
DEAN HOUSE PICCADILLY BRADFORD YORKSHIRE

INTERNATIONAL FEDERATION OF ASSOCIATIONS OF TEXTILE CHEMISTS AND COLOURISTS

1959 INTERNATIONAL CONGRESS IN LONDON

The first visit to England by the International Federation of Associations of Textile Chemists and Colourists will be made in 1959 when the *host member Society* will be The Society of Dyers and Colourists

The Congress will be held on
WEDNESDAY THURSDAY FRIDAY 16 17 18 SEPTEMBER 1959
with the Congress centre at

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During the Congress the social events will include a Dinner and Gala Ball in The Great Room, Grosvenor House, Park Lane, London
on
Friday evening 18 September 1959
The social events will also include tours and special attractions for the ladies

Already heavy advance reservations have been received for tickets and all interested are invited to communicate with —

THE GENERAL SECRETARY
THE SOCIETY OF DYERS AND COLOURISTS
DEAN HOUSE · 19 PICCADILLY · BRADFORD 1 · YORKSHIRE · ENGLAND

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1958 and pages 509–516 of the July 1958 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Recent Developments in Dyeing for the Carpet Trade — *W. Beal*

The Dyeing of Acrilan Acrylic Fibre — *H. R. Hadfield and M. Sokol*

The Latest Trends in the Development of the Field of Azoic Dyes — *M. Hückel*

Flameproofing of Textile Fabrics with particular reference to the Function of Antimony Compounds — *N. J. Read and E. G. Heighway-Bury*

COMMUNICATIONS

The Dyeing of Secondary Cellulose Acetate with Disperse Dyes
VII— A Comparison with their Solubility in Organic Solvents — *C. L. Bird*

Adsorption at Organic Surfaces—

III— Some Observations on the Constitution of Chitin and on its Adsorption of Inorganic and Organic Acids from Aqueous Solution
C. H. Giles, A. S. A. Hassan, (Miss) M. Laidlaw, and R. V. R. Subramanian

IV— Adsorption of Sulphonated Azo Dyes on Chitin from Aqueous Solution
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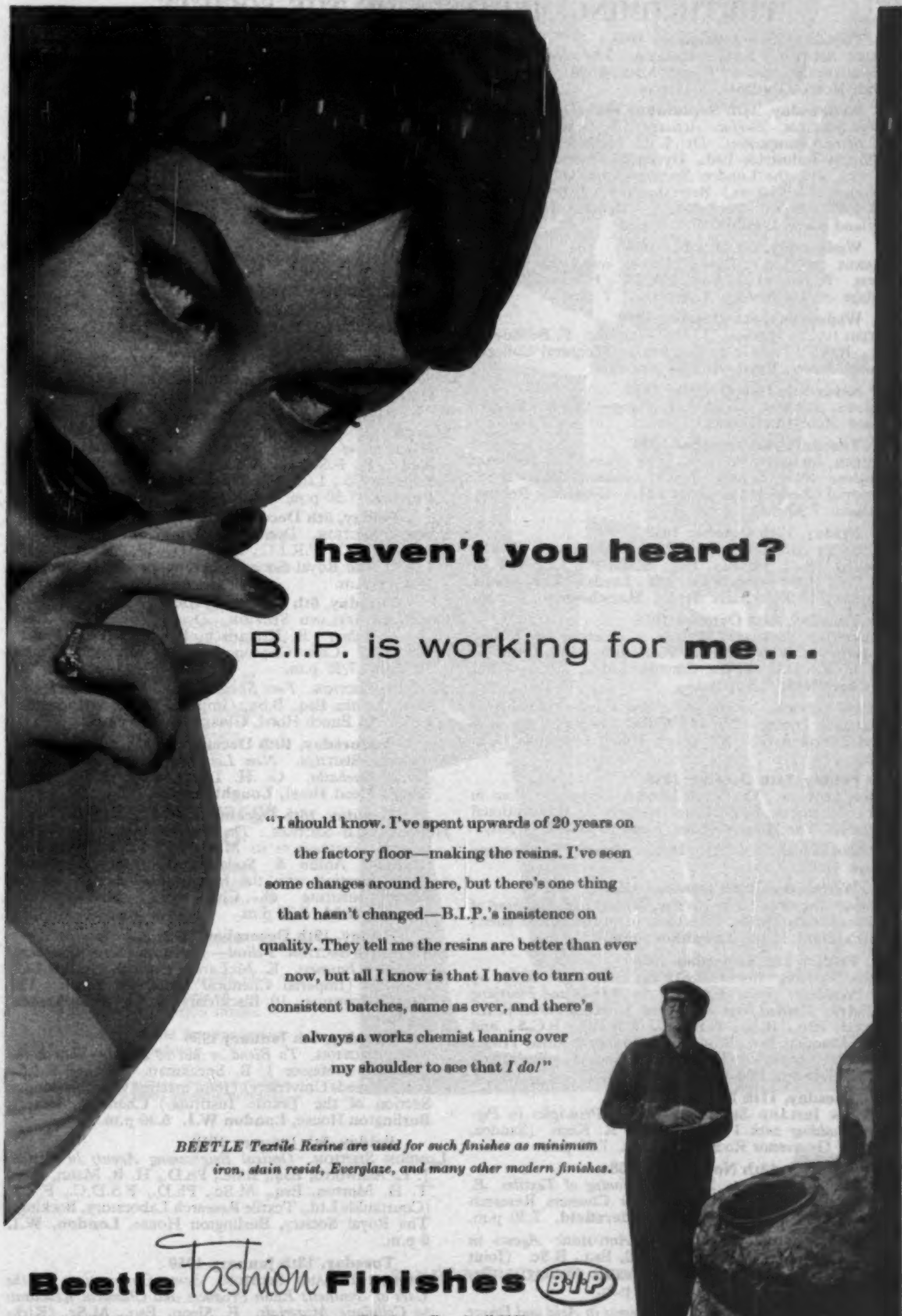
V— A Comparison of the Adsorption of Solutes by Cellulose and Chitin
C. H. Giles and A. S. A. Hassan

Unlevel Dyeing in Wool Velour Cloth — *F. J. Parker*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption — *C. H. Giles*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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 23rd September 1958

SCOTTISH SECTION. Ladies Evening. *The Good Housekeeping Institute and its Work.* Miss A. M. Kaye. St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 24th September 1958

LONDON SECTION. *Surface Activity: The General Concept and Some Consequences.* Dr. J. L. Moilliet (Imperial Chemical Industries Ltd., Dyestuffs Division). (Joint Meeting with the London Section of the Oil & Colour Chemists' Association.) Refreshments will be served at 6.15-6.45 p.m. in the Library. Mansion House, 26 Portland Place, **London W.1.** 7 p.m.

Wednesday, 1st October 1958

MIDLANDS SECTION. *Dyeing Unions containing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. College of Technology, **Leicester.** 7 p.m.

Wednesday, 8th October 1958

SCOTTISH JUNIOR BRANCH. *Colour Matching.* E. Bellhouse, Esq., B.Sc. (The Geigy Co. Ltd.). Technical College, George Street, **Paisley.** 7.30 p.m.

Saturday, 11th October 1958

MIDLANDS SECTION. Ladies Evening—Dinner Dance. Grand Hotel, **Leicester.**

Tuesday, 14th October 1958

NORTHERN IRELAND SECTION. *The Dyeing of Terylene/Cellulosic Fibre Blends.* J. G. Graham, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Friday, 17th October 1958

MANCHESTER SECTION. *Some Recent Advances in Textile Printing.* R. J. Hannay, Esq., B.Sc., F.R.I.C., F.T.I., F.S.D.C. (Brotherton & Co. Ltd., Leeds). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Tuesday, 21st October 1958

HUDDERSFIELD SECTION. *Methods of Assessing the Dyeing Properties of Wool Dyes.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Silvios Cafe, **Huddersfield.** 7.30 p.m.

SCOTTISH SECTION. *A Survey of the Various Methods of Continuous Dyeing.* Dr. H. Waibel (Badische Anilin & Soda Fabrik A.G.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Friday, 24th October 1958

LONDON SECTION. The Sixth London Lecture. *Colour in Art.* A. Lucas, Esq. (Chief restorer to the National Gallery). The Waldorf Hotel, **London W.C.2.** 7 p.m.

NORTHERN IRELAND SECTION. Dinner Dance. Woodbourne House Hotel.

Wednesday, 29th October 1958

MIDLANDS SECTION. *The Setting, Dyeing and Finishing of Banlon Knitted Goods.* (Lecturer to be announced later.) King's Head Hotel, **Loughborough.** 7 p.m.

Friday, 7th November 1958

LONDON SECTION. Investiture of the Chairman's Badge by the President. *Some Aspects of the Dyeing and Finishing of Fabrics Knitted from Bulky and Stretched Yarns.* S. M. Jaeckel, Esq., B.Sc., A.R.C.S., A.R.I.C., F.C.S., and D. L. Munden, Esq., B.Sc. (The Hosiery & Allied Trades Research Assoc.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 11th November 1958

NORTHERN IRELAND SECTION. *General Principles in Pigment Padding with Vat Dyes.* Dr. R. Kern. (Sandoz, Basle). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Tuesday, 18th November 1958

HUDDERSFIELD SECTION. *The Dry Cleaning of Textiles.* E. J. Davies, Esq., M.Sc. (Dyers & Cleaners Research Organisation). Silvios Cafe, **Huddersfield.** 7.30 p.m.

MIDLANDS SECTION. *The use of Anti-static Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Joint meeting with the Nottingham Textile Society.) Gas Board Theatre, **Nottingham.** 7 p.m.

SCOTTISH SECTION. *New Levelling Agents in Acid and Direct Dyebaths.* G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 19th November 1958

SCOTTISH JUNIOR BRANCH. *Some Textile Dyeing and Drying Machines.* K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). (This lecture will be illustrated by means of a film show.) Technical College, George Street, **Paisley.** 7.30 p.m.

Thursday, 20th November 1958

LONDON SECTION. *Developments in Modern Dry Cleaning Techniques.* E. J. Davies, Esq., M.Sc. (Dyers & Cleaners Research Organisation). (Joint meeting with the London Centre of the Guild of Dyers & Cleaners.) Institute of Journalists, 2-4 Tudor Street, **London E.C.4.** 7.15 p.m.

Friday, 21st November 1958

HUDDERSFIELD SECTION. Annual Dinner. Princess Cafe, Northumberland Street, **Huddersfield.**

MANCHESTER SECTION. Ladies Evening. College of Science and Technology, **Manchester.** Further details later.

Monday, 24th November 1958

HUDDERSFIELD SECTION. Invitation from The Halifax Textile Society—Joint meeting at The Alexandra Hall, **Halifax.** 7.30 p.m.

Tuesday, 2nd December 1958

SCOTTISH JUNIOR BRANCH. *Some of the Principles and Practices of Textile Printing.* A. Howarth, Esq., A.M.C.T., F.S.D.C., A.T.I. (The Seedhill Dyeing & Printing Co., Ltd.). Technical College, George Street, **Paisley.** 7.30 p.m.

Friday, 5th December 1958

LONDON SECTION. *Dyeing of Furs.* J. L. Stoves, Esq., M.Sc., Ph.D., F.R.I.C., F.C.S. (C. W. Martin & Sons Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 9th December 1958

NORTHERN IRELAND SECTION. *Dyeing Unions Containing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Fast Shades on Wool-Cellulose Unions.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 10th December 1958

MIDLANDS SECTION. *New Levelling Agents in Acid and Direct Dyebaths.* G. H. Lister, Esq., B.Sc., Ph.D. King's Head Hotel, **Loughborough.** 7 p.m.

Tuesday, 16th December 1958

HUDDERSFIELD SECTION. *The Development of a Dyestuff from its Synthesis to its Marketing.* K. Roessler, Esq. (Badische Anilin & Soda-Fabrik A.G., Germany). (Joint meeting with the Huddersfield Section of the Royal Institute of Chemistry.) Silvios Cafe, **Huddersfield.** 7.30 p.m.

Friday, 19th December 1958

MANCHESTER SECTION. *Felisol—An International Guarantee of Colour Fastness.* K. McLaren, Esq., B.Sc., F.R.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Tuesday, 6th January 1959

LONDON SECTION. *To Blend or not to Blend—That is the Question.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Leeds University). (Joint meeting with the London Section of the Textile Institute.) Chemical Society, Burlington House, **London W.1.** 6.30 p.m.

Friday, 9th January 1959

LONDON SECTION. *Optical Brightening Agents in Action.* J. L. Ashworth, Esq., B.Sc., Ph.D., H. B. Mann, Esq., T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C., F.T.I. (Courtaulds Ltd., Textile Research Laboratory, Bocking.) The Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 13th January 1959

NORTHERN IRELAND SECTION. *Some Observations in the Uses of Synthetic Resin Products and Chemical Reactants to Cellulosic Materials.* F. Sloan, Esq., M.Sc. (Kirkpatrick Bros. Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

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THE JOURNAL OF THE

Society of Dyers and Colourists

Volume 74 Number 8 AUGUST 1958 *Issued Monthly*

Proceedings of the Society

Fast-to-washing Dyeing of Wool Yarn

J. F. GAUNT

Meeting of the West Riding Section held at the Hotel Metropole, Leeds, on 28th November 1957, Mr. G. E. Styan in the chair

The dyeing of wool yarn with dyes other than the level-dyeing type has become important only since about 1950, but it is now possible to dye almost all colours fast to the S.D.C. No. 2 Washing Test (*B.S.* 2684) and yet sufficiently level for knitting in solid-shade garments. The requirements of the various trades using fast-dyed yarn are considered, and an outline is given of the developments in dyes and dyeing machines leading to the present-day position. Advances have been made in dyes, dyeing machines, and dyeing methods, and these have been complementary. These aspects are discussed separately, and attention is drawn to complications which have arisen as a result of the adoption of new methods.

Introduction

The dyeing of fast-to-washing colours on wool yarn, as the term is understood today, means the production of a very wide range of colours which withstand repeated hand-washing with little or no alteration of colour or bleeding into adjacent material. The standards which are required today are higher than at any previous date, but the textile industry has always been searching for better quality at all stages of processing, and as developments take place in the fields of dyes, dyeing techniques, and dyeing machines, it is certain that even better fastness will become practicable in due course.

The growth in the importance of fast yarn-dyeing has been very rapid. Prior to 1950, it was virtually non-existent, nearly all woollen and worsted yarn being dyed with level-dyeing acid dyes. This development has not been due to any single factor, but has arisen from a combination of improvements in various directions, and it is interesting to consider the background of these changes.

Old prints show that wool yarn has been dyed in hank form for some hundreds of years, but the introduction of yarn-dyeing on a large scale is comparatively recent. The first patent for a dyeing machine was granted in 1882[1]. This machine, the Obermaier type SA, was of the design still widely used for dyeing loose material, for which it was originally designed, but attempts were soon made to use it for yarn-dyeing by packing the hanks inside the material container[2]. At that period, however, the most commonly used method for dyeing yarn was to suspend the skeins over wooden poles which rested on the sides of an open vat, the skeins being constantly lifted out of the liquor and altered in position by two men, one at each side of the vat. According to Horsfall and Lawrie[3], dyeing by this method was still in use in 1927 both for worsted yarn and for woollen carpet yarn, although presumably by this date dyeing by hand was rapidly being ousted by machine-dyeing. An excellent description of the procedure is given by Isles[4], but whatever may be the virtues of the method as regards the production of dyeings of acceptable levelness from natural colouring matters and the first synthetic dyes, which generally had only poor level-dyeing properties, there is no doubt that the method was very slow and laborious, and that great care and experience were necessary to avoid unlevelness on the one hand and deterioration of the condition of the yarn on the other. For these reasons, it was much more common for coloured yarn to be produced by dyeing as loose wool, for not only did this give more level results owing to the mixing which took place during carding, but the yarn was in much better condition and larger blends of the same colour were practicable. Loose-wool dyeing was in use on a large scale for the production of coloured worsted yarns for some time after 1920, and this method of dyeing is, of course, still used to a great extent in the production of woollen yarns.

Shortly after the advent of the Obermaier machine in 1882, other types of machine were specially developed for hank-dyeing, but most of these early types of machines are now obsolete and indeed almost forgotten. The only type still in use is the Klauder-Weldon machine, which

works on a completely different principle from that of other wool-yarn dyeing machines. In the Klauder–Weldon, the yarn is moved through the dye liquor, instead of the liquor being circulated through the yarn, as is the case with all other present-day yarn-dyeing machines. The sticks from which the yarn is supported in the Klauder–Weldon machine are arranged radially within a large rotating wheel and dip into the dye liquor at the lower part of the revolution of the wheel. These machines, although now almost extinct in this country, are still widely used in America, where the latest types are constructed of stainless steel. There is much to commend the principle of the Klauder–Weldon machine, in which channelling of the liquor, so common and troublesome in many other forms of dyeing machine, is minimised by the hanks being brought into contact with the liquor at each revolution of the wheel. The great objections to such machines are that they are more difficult to operate, and appreciably more costly to maintain, than other designs.

Other machines which were used for yarn-dyeing in the early years of this century were the Partridge and Lorrimer machines [5]. The Partridge machine, at one time widely used for dyeing carpet yarn, consisted of a rectangular frame with two sets of dye sticks at opposite ends of the frame, with the hanks suspended between the opposite sticks. The frame was pivoted so that it could be rotated on a horizontal axis at right-angles to the skeins. During the dyeing process the whole frame was slowly raised and lowered in the dye-vat and was given a quarter of a revolution at the end of each cycle. The machine was inconvenient to load and unload and had to be operated at a very high liquor ratio, with the result that there was heavy consumption of steam and water, and it is unlikely that any examples of this machine remain in existence. The Lorrimer machine was a mechanised version of the old hand-dyeing process already mentioned, the skeins being hung from wooden sticks held in a frame which was raised and lowered about 8–10 in. at intervals of 1–2 min. throughout the dyeing operation. A hydraulic ram was often used for raising and lowering the frame. Here, again, it is probable that this type of machine is now completely obsolete.

In Germany, machine design followed other courses. The Esser machine, a description of which is given by Bolton [6], may be regarded as the prototype of the present-day Continental hank-dyeing machines. The material carrier had an upper and a lower set of dye sticks, which held the hanks of yarn, and the liquor was circulated by means of a propeller. Attempts were made to employ alternative methods for circulating the liquor through the yarn, e.g. the Obermaier type GA machine, in which the material carrier, of a similar type to that in the Esser machine, was moved up and down in the dye-vat by means of a reciprocating motion, no propeller being used. Machines of this type, of up to 100 kg. capacity, were constructed as recently as 1921 [2].

The machine which is now in most general use in this country for dyeing woollen and worsted yarn in hank form is the Hussong, introduced from America during the first few years of this century. Such machines have proved their value on a wide variety of wool qualities and yarn counts, and machines of this design in a great range of sizes are to be found in practically every dyehouse where woollen and worsted yarns are dyed. The first machines were made of wood, but since 1931 there has been a rapid development in the use of stainless steel for all types of dyeing machine.

During the period in which the improvements in dyeing machines outlined above have taken place, the development of an adequate range of wool dyes of good level-dyeing properties has also occurred. It is, perhaps, difficult to appreciate that even as recently as twenty-five years ago it was impossible to produce a good range of bright colours possessing the standards of fastness to light and wet processing which are now regarded as commonplace, whilst it is less than sixty years since the discovery of the level-dyeing acid dyes, which are now being superseded by faster types. The first synthetic dyes, produced a century ago, were either basic dyes or chrome dyes. The former, although bright in colour, have poor level-dyeing properties on wool, where their light fastness is quite unacceptable by modern standards. The first chrome dyes, although possessing good fastness properties, were difficult to dye level on wool and were all dull in shade. The level-dyeing acid dyes, which for many years have been used to a greater extent on yarn than any other class of dye, are of considerably later origin, as shown in Table I [7]. These dyes, many of which are still in very wide use on yarn, led to the rapid growth of yarn-dyeing around the beginning of this century. Dyes of the milling acid type and the important group of metal-complex dyes are all of much later date.

TABLE I

Dates of Discovery of Level-dyeing Acid Dyes

Acid Magenta	C.I. Acid Violet 19	1877
Fast Red A	C.I. Acid Red 88	1877
Light Green SF	C.I. Acid Green 5	1879
Tartrazine	C.I. Acid Yellow 23	1884
Patent Blue V	C.I. Acid Blue 3	1888
Naphthalene Green V	C.I. Acid Green 16	1889
Acid Violet 6B	C.I. Acid Violet 17	1890
Alizarine Saphirol B	C.I. Acid Blue 45	1897
Cyananthrol R	C.I. Acid Blue 47	1901
Amido Naphthol Red G	C.I. Acid Red 1	1902
Sulpho Rhodamine B	C.I. Acid Red 52	1903
Xylene Light Yellow 2G	C.I. Acid Yellow 17	1908

In the light of the above brief historical background, the growth of the use of fast-to-washing dyes in place of level-dyeing acid dyes will now be considered.

Trades for which Wool Yarn is dyed

The trades for which woollen and worsted yarns are dyed are as follows—

(1) Hosiery (machine-knitting and hand-knitting)

(2) Carpets and rugs

(3) Upholstery fabrics
(4) Woven apparel fabrics.

1. HOSIERY (MACHINE-KNITTING AND HAND-KNITTING)

With comparatively few exceptions, all knitted garments, whether produced by machine-knitting or by hand-knitting, are made from clean-scoured dyed yarn. There is some dyeing of knitted fabrics as either tubular fabric or half-hose, and also as underwear and outerwear, where the economic advantages of being able to keep an undyed stock of material until as late a stage as possible, and the possibility of giving rapid delivery of any desired colour, are important. For some high-quality knitted garments, top-dyed yarn is used, and the garments are knitted from the oily yarn, scouring taking place after knitting. Apart from these types of yarn, however, it is universal to knit from yarn which has been dyed and scoured before knitting. Until about 1950, it was generally considered that colours of good fastness to washing had to be produced by top-dyeing, in order to obtain the requisite high degree of levelness from the milling acid and chrome dyes needed for this standard of fastness. A considerable weight of machine-knitting yarn was hank-dyed with level-dyeing acid dyes, but the washing fastness of these, particularly in deep colours, left a great deal to be desired. During the last few years, there has been a transformation in the methods used for dyeing these yarns, and dyes of good washing fastness are now yarn-dyed on a large scale. The former distinction between dyeing in slubbing or top form to obtain good washing fastness and yarn-dyeing only with level-dyeing acid dyes no longer applies. Of the very large weight of machine-knitting yarn dyed by Patons & Baldwins Ltd., all colours, apart from an insignificant proportion, are now dyed fast to the S.D.C. No. 2 Washing Test*.

In the case of hand-knitting yarn, similar considerations formerly applied, with the exception that it was the custom with reputable firms to slubbing-dye an even greater proportion of hand-knitting yarn than of machine-knitting yarn. It is, of course, desirable to maintain stocks of hand-knitting yarn as flexible as possible, and this is facilitated by yarn-dyeing the solid shades. Nevertheless, for good-quality hand-knitting yarns the change from slubbing dyeing to yarn dyeing is accepted only when it has been proved that it can be made without any sacrifice of fastness or of other desirable properties of the yarn.

These two classes of machine-knitting and hand-knitting yarn are probably the most important sections of the trade where the fast dyeing of yarn is employed. Naturally, the introduction of these fast-to-washing dyes has led to a number of technical problems, and discussion of these forms the basis of this paper.

2. CARPETS AND RUGS

In the carpet industry it is not uncommon for very large blends, often as large as 20,000 lb., of exactly the same shade of yarn to be required for plain carpets, and in these cases dyeing is carried out in the form of loose wool. It is usual to employ dyes of good fastness to wet processing and to light, and any slight unevenness at the loose-stock stage is of no consequence, since carding and spinning give a level result. In general, however, the carpet industry uses yarn dyed in hank form, and for this level-dyeing acid dyes are by far the most important class. Hussong machines, often of very large capacity, up to 2000 lb., and now coupled so as to give batches of up to 9000 lb. per shade [8], are employed. The level-dyeing acid dyes have a standard of light fastness which is generally adequate, and they permit the very accurate matching of colour, which is a prime requirement in the carpet industry. Their wet fastness in full depths is poor, and during the last few years there has been a movement towards the application of dyes of better washing fastness which will withstand domestic shampooing. Angus [9] has referred to the difficulty of obtaining level results on carpet yarn, and this is somewhat surprising in view of the coarse wools used in carpet yarn, since it is generally found with wool dyeing, just as with viscose rayon [10], that as the fibre diameter decreases the rate of dyeing increases. It is likely that this difficulty of obtaining level results with fast dyes on carpet and rug yarn arises from other causes peculiar to this type of yarn, and these will be discussed later.

3. UPHOLSTERY FABRICS

For this important section of the textile industry, yarn dyeing is carried out almost exclusively with level-dyeing acid dyes, although there is now some movement towards top-dyeing in order to obtain fastness to shampooing.

4. WOVEN APPAREL FABRICS

In this country, it is usual for woollen and worsted apparel fabrics to be either piece-dyed or else dyed as slubbing for the worsted trade and loose wool for the woollen trade. Dyeing prior to spinning enables dyes of the maximum fastness properties to be applied and also ensures that the whole of the yarn even in a very large blend is of precisely the same colour. Yarn dyeing would demand smaller unit weights, with the danger of slight colour variation between blends. However, for certain special purposes, e.g. threads for stripes and other effects, yarn is occasionally dyed on cheese or cone, mainly where the weights are of the order of 100 lb. or less, which would hardly justify dyeing as loose wool or slubbing and the subsequent spinning of a small lot. Such striping threads are generally used in broken designs, and

* For the hosiery trade, where coloured yarn is often knitted against white, it is important that there should be no staining whatever into the white material during washing. Consequently, this is the most important aspect when evaluating washing tests, and the bleeding into white must have a figure of 4–5 or 5. Staining of cotton during washing is of no importance as such, but frequently is a good indication of any potential dangers of marking-off on to cotton or viscose rayon underwear as a result of perspiration during wear. It is, therefore, advisable to aim at a minimum standard of 4 on the S.D.C. grey scale for assessing staining. With the growing use of nylon underwear, it is often useful to incorporate a small piece of nylon in the washing test as a safeguard against any danger of staining nylon as a result of perspiration.

The colour of the wool, whilst perhaps not quite so important as the absence of any staining of adjacent white material, is nevertheless very critical. Only under extreme circumstances would material be acceptable if the change in colour were greater than 3–4 on the S.D.C. grey scale, and even then the change would have to be on tone.

thus the levelness of colour is not as vital as in a solid-shade fabric. It should be pointed out, however, that the position with regard to yarn dyeing is quite different on the Continent, where it is common to dye singles worsted yarn for suiting cloths and to ply the yarn after dyeing. Such a practice appears to have a number of advantages, and one would expect it to become more common in this country.

Colour and Fastness Requirements of Hosiery Yarns

Yarn to be used for machine-knitting or hand-knitting must possess the following properties, which are arranged in order of decreasing importance—

(1) Levelness of colour
(2) Good handle
(3) Washing fastness
(4) Light fastness
(5) Fastness to subsequent processing, e.g. bleaching or shrink-resisting.

1. LEVELNESS OF COLOUR

When dyed yarn is knitted into a solid-shade garment, particularly on a single-feed machine, or when it is hand-knitted, the yarn actually being knitted forms the whole of the fabric and is not divided into warp and weft components as in a woven cloth. Consequently, any local variation in depth or in hue within a hank of hosiery yarn is quickly laid alongside another part of the material and is thrown into relief. Similarly, if there is any variation in colour between the hanks or cones of yarn from the same dyed lot, the extremes are sooner or later brought together when the end of one package is knitted alongside the beginning of the next. Knitting is, therefore, the most critical test of levelness of colour, and the nearest comparison to it in woven fabrics is in a plain carpet, where similar considerations apply. A colour variation along the warp of a woven suiting fabric would stand considerably less danger of being noticed. In any form of knitting yarn, levelness of colour is of paramount importance. If this requirement is not met, the yarn is practically valueless.

2. PRESERVATION OF HANDLE

The types of wool used for knitting yarn are chosen to be as soft as possible consistent with good wearing properties, and care is taken to use spinning and folding twists directed to the same end. Dyeing processes which would give the yarn a harsh or brittle handle cannot be tolerated. Because of the relatively slack twist, hosiery yarns are more prone to felting and end-breakage during dyeing than are other types of yarn. Nevertheless, no felting can be accepted during a commercial dyeing process, not only because of the adverse effect on the appearance and the properties of the yarn, but also because this would lead to difficulties during the subsequent winding process. Great care must be taken to avoid any disturbance of the skeins during dyeing, and this naturally has a bearing, not only on the dyeing process itself, but also on the type of dyeing machine used.

3. WASHING FASTNESS

Not only must the wool maintain its colour throughout the life of the garment, but also (and probably more important) there must be no bleeding of one colour into another. The hot alkaline processes used for cellulosic fibres would ruin the wool fibre itself, irrespective of any change in the colour of the dyes present, and in practice milder conditions are used for washing wool garments. In general, it has been found that the S.D.C. No. 2 Washing Test (*B.S.* 2684) represents a satisfactory minimum level of washing fastness*. This gives a good indication of the behaviour of the garment when washed by hand by the methods recommended by the makers of the soap and detergent products used in the household. Should the public demand a standard of washing fastness adequate for garments to be washed at higher temperatures, e.g. in washing machines, very severe difficulties would be introduced. Not only would the wool itself have to be treated in some special way to avoid felting, but also there are at present no dyes available for obtaining a higher standard of washing fastness for many of the more popular colours. Whilst the chrome dyeings withstand more stringent washing conditions, the colours are without exception dull. Full depths of nearly all existing acid milling dyes have a tendency to bleed under conditions more drastic than the S.D.C. No. 2 Washing Test, thereby rendering it impossible to produce these heavy bright colours with a higher degree of fastness.

4. LIGHT FASTNESS

Light fastness is often one of the most difficult requirements to fulfil. Many of the traditional hand-knitting colours are very bright, being based on the earliest synthetic basic dyes, whose main attribute was brightness rather than light fastness. More detailed reference will be made to this later in this paper, but in general it can be said that, given a light fastness of 3–4 on the *B.S.* 1006 scale, there are unlikely to be serious numbers of fading complaints on yarn used for outerwear, at any rate as regards conditions in this country.

5. FASTNESS TO BLEACHING AND SHRINK-RESISTING

Because of the demand for brightness of colour in hosiery yarns, it is frequently necessary to bleach after dyeing. This introduces complications, for there is often some change in hue, and allowance has to be made for this when the dyer passes the shade. Bleaching before dyeing is, however, virtually useless, as any brightening arising from the peroxide is lost during the subsequent dyeing process. It is therefore necessary to select dyes with good fastness to bleaching. Similar considerations arise with regard to shrink-resisting, which is usually carried out after dyeing. Many dyes are affected by the chemicals used in the commercial shrink-resisting processes, so that dyes must be selected of adequate fastness to avoid the colour becoming unlevel.

* See footnote on p. 571.

Development of Hosiery Yarn Dyeing

It has already been mentioned that for many years it was generally accepted that the only group of dyes which could be applied sufficiently level for solid-shade knitting were the level-dyeing acid dyes, and that dyes of good washing fastness could be applied only with a standard of levelness suitable for striping threads. Demands for a better standard of washing fastness have led to intensive work in many directions, since the problem of applying such dyes is far from easy to solve. The difficulty arises primarily from the fact that, whereas level-dyeing acid dyes have excellent migrating properties, this property is not shared by any other class of dye. This absence of migration of nearly all dyes of good washing fastness is of such importance that it can hardly be overemphasised. Care has to be taken, therefore, to ensure that the dye is taken up evenly from the beginning of the dyeing process, and to achieve this work on applying these fast colours has followed three interdependent lines— (1) machine design, (2) dye selection, and (3) dyeing methods. These will now be discussed in detail.

1. MACHINE DESIGN

The Hussong machine has been the standard design for hank-dyeing in this country for almost fifty years. It is simple in design and easy to operate and maintain. With level-dyeing acid dyes it has been used to produce practically all colours with little or no difficulty. It is still the most important type of machine, but there is no doubt that its limitations are becoming more obvious as the changeover to the use of fast-to-washing dyes continues. Since an even strike is essential with the fast dyes, there must be uniform and adequate circulation of the dye liquor throughout the material. In the Hussong, the yarn is usually packed fairly tightly along the sticks, and furthermore it is common practice for the depth of the machine to be such that the distance between the top of the stick and the false bottom is less than the length of the skeins. It is not unusual for machines used for dyeing yarn in 72-in. reel to have less than 24 in. between the top stick and the perforated false bottom. Consequently, the hanks are in a crumpled form, and the actual path followed by the liquor must be highly problematical. This is not of particular importance with level-dyeing acid dyes, since, although the initial uptake of the dye may be highly unlevel, boiling for an hour or more in an acid liquor gives an acceptable degree of levelness. This does not occur with the fast dyes. With a Hussong it is therefore necessary to alter the dyeing conditions, viz. either the mechanical behaviour of the dye liquor or else the composition of the dyebath, so that the dye is absorbed evenly, but this is frequently difficult. The problem of improving the mechanical conditions of the flow in a Hussong machine is far from easy. Merely to increase the rate of flow by fitting a more powerful propeller is unsatisfactory, since the yarn is packed to a greater or less degree and there is no clear passage for the liquor, which consequently merely channels to an even greater extent than with the lower rate of flow. This is accompanied by undesired turbulence of the liquor and by most undesirable felting of the yarn. In addition, Hussong machines, particularly those of large size, have a "dead spot" at the end of the machine away from the propeller, and even with an increased rate of flow there is a great difference in the behaviour of the liquor at the two ends of the machine. The other alternative of altering the composition of the dye liquor will be discussed later when dyeing methods are considered.

In view of the above, considerable attention has been given to alternative methods of dyeing. Continuous dyeing, such as is used for cotton warps in the Lancashire trade, has not yet been found applicable to woollen or worsted yarn, and it is unlikely that any method suitable for dyeing hosiery yarn will appear for some considerable time, because of the fatal results of even the slightest "ending".

Machine developments have taken two main directions— (*a*) package dyeing and (*b*) hank dyeing—each of which possesses distinct advantages.

(*a*) *Package Dyeing*

From a production point of view, package dyeing is very attractive. The sequence of operations is as follows—

(i) Wind on to dyeing package from spinning bobbin or twist bobbin
(ii) Scour, rinse, and dye in dyeing machine
(iii) Remove gross water by hydroextracting or with compressed air
(iv) Dry in a cupboard dryer or a "rapid dryer"
(v) Wind on to whatever form of package is required by the customer.

Throughout the above processes, the yarn is not disturbed and there is consequently no danger of felting, cutting, or fraying, which is of considerable advantage with yarn of very fine count. In the case of yarns with a high twist factor, where cockling would be so serious as to necessitate the expensive and laborious additional process of setting before hank-dyeing, the yarn can be package-dyed without any pretreatment. Another advantage of package dyeing is that, because of the uniform winding tension and the uniform rate of flow of liquor through each package, less danger of channelling of the liquor should occur, so that level-dyeing would be expected to be less of a problem than with a Hussong machine.

(*b*) *Hank Dyeing*

In spite of the important advantages of package dyeing, in fact there is only a relatively small amount of hosiery yarn dyed on cheese or cone in this country. It is much more common to dye in hank even though the sequence of operations—

(i) Reel
(ii) Scour, usually through a tape machine
(iii) Hank-dye
(iv) Hydroextract
(v) Dry through a tunnel dryer
(vi) Rewind

is more complicated and involves the slow reeling and hank-to-package winding processes (about 200–250 yd./min. as against up to 1,000–1,200 yd. per min. for package-to-package winding), and also more danger of yarn damage at the various stages. Whilst undoubtedly requiring more handling than package dyeing, hank dyeing has the following advantages—

(A) Hank scouring, in which several sets of squeeze heads and underwater squeezes are used, is more thorough and reliable and certainly removes soiling and dirt marks more efficiently than package scouring, in which it is possible only to pass a detergent liquor through the yarn without any form of squeezing. This problem of scouring on package is more acute with woollen-spun yarns and with worsted yarns spun in oil on the Bradford system than with dry-spun worsted yarns.

(B) In package dyeing, the yarn is necessarily wound under some tension, and the yarn is distinctly flat and lacking in bulk as compared with the same quality of yarn dyed in hank. This is the case even with fine counts, but is even more noticeable in heavier counts, e.g. in the type used in hand-knitting, where an added disadvantage is that the kink in the yarn at each intersection over the traverse beneath it shows up clearly. A hand-knitting yarn dyed on cheese or cone would be quite unacceptable.

(C) It is virtually impossible to ensure that there is no fine dirt or other insoluble matter introduced into a dye liquor from the dyes, auxiliary products, Glauber's salt, etc. In package dyeing this tends to be concentrated at the inner and outer surfaces, whereas in hank dyeing it is more likely to remain suspended in the liquor or, at the worst, spread over a much larger area and hence at a much lower concentration. Similarly, only dyes of good solubility are suitable for package dyeing, whereas a wider selection of dyes is available for hank dyeing.

(D) Whilst modern dyeing techniques have reached a high level of reliability, it is very useful for the yarn to be examined after dyeing and drying. With yarn in hank form this thorough examination, even though slow and somewhat tedious, is not difficult, but when the yarn is in package, it is impossible to see the levelness of colour without back-winding. This, of course, eliminates one of the attractive features of package dyeing, but failure to do this, at any rate with part of each batch, means that the dyer has to take it for granted that the yarn is level and on shade throughout.

(E) Hank drying is simple and straightforward, and there are many types of dryer which can be used. With packages, the yarn must be dried in either a cupboard dryer, which may take up to 24 hr. after hydroextracting, or an expensive rapid dryer, which frequently necessitates the use of motors of up to 70–90 h.p. for drying batches of 200 lb.

(F) The usual form of spindle on which dyeing packages are wound is made of stainless steel. To obtain the best economic advantages from package dyeing, the yarn must be wound direct on to the dyeing package from the spinning or twisting bobbin, and thus the stock of undyed yarn must be kept either on spinning packages or on dyeing packages. In either case there is a considerable amount of capital tied up in bobbins, which could be of the order of 10s. 0d. per lb. of yarn in stock, and hence this could readily amount to some hundreds of thousands of pounds in the case of a large firm.

In view of the above, there is much to be said for a reliable hank-dyeing machine of sound design and construction. Such a machine allows yarn of virtually all counts to be dyed with the possible exception of certain fine singles yarn, and it certainly produces a loftier yarn of better handle and appearance than does package dyeing.

It appears that the improvement in flow cannot be achieved by a single-stick machine, since on the upward flow of the liquor there is excessive movement of the hanks and hence deterioration in condition. Furthermore, at the relatively high pH values of 6 and upwards, which are sometimes necessary with certain types of fast acid dye, there is objectionable floating of yarn, which is rarely observed with ordinary acid dyes. The trouble manifests itself by the yarn rising to the surface of the liquor, thereby almost completely preventing circulation of the latter. Occasionally this can take place suddenly, and instances have been known where floating of yarn has occurred so violently that the lid of the machine has been torn loose from its clamps.

The obvious solution to the problem of skein dyeing is, therefore, to use machines with two sets of sticks, so that the yarn is supported at top and bottom, and hence in both directions of flow of liquor. Such machines have been in use on the Continent for a number of years, the first Obermaier machines of this type being delivered in 1929–1930. These machines have a removable material carrier similar in principle to that of the Hussong, but in earlier models the sticks were in sliders on the inside of the machine and were loaded through a door at the end of the dye-vat [2]. This latter type of machine has since been further developed in Italy.

Developments in the details of design have taken place constantly, e.g. within the past two years such machines have been modified to operate at temperatures above 100°C. In addition to various Continental designs, double-stick machines are now being developed in this country by one or two machine makers, and it is likely that the principle will gain increasing acceptance as the advantages become more widely appreciated—

(i) The machine is sufficiently deep to take the yarn at the full length of the skein, which is consequently not crumpled as in a Hussong. Furthermore, the yarn is supported at all stages, and the liquor is free to circulate between and through the skeins to an extent which is impossible in the pack form in which the yarn finds itself in a Hussong. The bubbles of gas giving rise to floating of yarn in a Hussong are able to disperse steadily.

(ii) It has already been mentioned that a high rate of liquor flow is necessary when applying fast dyes and that this is impossible in a Hussong machine. In a double-stick machine, as a result of the better method of supporting the skeins the rate of liquor flow can be greatly increased, and it is not uncommon to find motors of at least twice the size of those which would be fitted to a Hussong of similar capacity.

(iii) The latest types of double-stick machines have material carriers in which the yarn is completely enclosed. There is, therefore, no danger of skeins at the sides of the load, and particularly at the corners, being "blown" about. Because of the generally straighter condition of the hanks during dyeing, the yarn is in much better condition, and extensive tests have shown that yarn dyed in a double-stick machine is more lofty, less matted, and more easily wound than yarn of the same quality dyed by a similar process in a Hussong machine.

(iv) The difficulty of ensuring adequate liquor flow throughout a Hussong machine is largely overcome in modern double-stick machines by fitting larger motors and propellers, but to obtain even better and more uniform flow, various machine makers have adopted curved or sloped vat bottoms, so as to direct the liquor into places which would otherwise tend to be "dead spots". Should the liquor flow be found too great for any particular quality of yarn, throttles operated from outside the vat are fitted, so that the flow can be reduced as desired.

(v) As in the case of a Hussong machine, double-stick machines give yarn which is appreciably more lofty than the same yarn dyed on cheese or cone.

(vi) These machines can be used either individually or coupled to give large batches of yarn of identical shade.

Whilst the double-stick machine is somewhat less convenient than the Hussong to load and unload owing to the more elaborate construction of the material carrier with its sides and rigid perforated base, in practice this is not a serious drawback. With the increasing complexity of modern dyeing processes, it is becoming more common to fit automatic control mechanisms, particularly adjustable liquor-flow-reverse gear and temperature-control apparatus, to these machines, and it is expected that such mechanisms will become even more important.

To summarise the position on dyeing machines for obtaining fast colours on wool yarn, a higher and more uniform rate of flow is necessary than can be obtained from the conventional single-stick hank-dyeing machine. For most counts of yarn there would appear to be a good case for the double-stick hank-dyeing machine. In general, as the counts of the yarn become coarser, so do the advantages of hank dyeing become greater; and conversely, the advantages of package dyeing increase as the counts become finer.

2. DYE SELECTION

In order to obtain fastness to the S.D.C. No. 2 Washing Test, level-dyeing acid dyes can rarely be used. In depths up to 0·5% they do not bleed into adjacent white material, and for some purposes the slight loss in depth is not objectionable. It is of interest to note that the fastness figures given by most dye manufacturers in their pattern cards can be very misleading, in that only one figure for washing fastness, i.e. that at "standard depth", is included. Pale shades of acid dyes have good washing fastness, and heavy depths of milling acid dyes frequently have only moderate washing fastness, even though the former may be given a low figure and the latter a high figure. Because of their level-dyeing properties, acid dyes are abandoned with some reluctance on the part of dyers, and any auxiliary product which would improve the wet fastness without seriously altering the colour would be just as useful with acid dyes on wool as such products have been with direct dyes on cellulosic fibres.

In the absence of such a product, it is thus essential to use dyes other than level-dyeing acid dyes for any depth greater than 0·5%, and current indications are that acid dyes will soon become completely obsolete for hosiery-yarn dyeing, even in pale colours. Many alternative types of dyes have been examined, but, with the exception of the fast acid dyes, the chrome dyes, and the metal-complex dyes, all have had to be rejected. The wide range of vat dyes is of little value on wool because of poor exhaustion, and in spite of the brightness of colour which can frequently be obtained, the level-dyeing properties are not good enough for yarn dyeing. The solubilised vat dyes of the Soledon (ICI) and Indigosol (DH) types are a particularly tantalising group. They can be applied to wool to a reasonable degree of exhaustion and levelness, but unfortunately there appears to be no satisfactory method of converting the solubilised leuco compound to the parent vat dye. All the usual regenerating agents, e.g. nitrous acid, dichromate, persulphate, and hydrogen peroxide, react preferentially with the wool, so that at least part of the leuco compound remains on the fibre, giving a dull colour of poor light fastness. The three classes of dye mentioned above will now be discussed individually—

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Because of the demand for bright colours in the hosiery trade, extensive use is made of the milling acid dyes. These vary very widely in their wet fastness properties from such ranges as the Supramines (FBy, FH) and Xylene Fast P (S) dyes, which are a compromise between wet fastness and level dyeing, to dyes which will withstand heavy milling. A different principle is adopted in selecting milling acid dyes (and also chrome dyes) for matching a shade from that adopted when using acid dyes. With the latter, virtually all colours are produced from the commonly used red, yellow, and blue dyes. With the milling acid dyes there is more tendency for selective dyeing (skitteriness) of individual dyes, and if compound shades were produced with a combination of red, yellow, and blue dyes, the

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result would be unattractive. It is, therefore, common practice to use a wide range of milling acid and chrome dyes, each as near as possible to that of the required colour. Thus, one would normally use a bright greenish yellow, e.g. Milling Yellow H5G (C.I. Acid Yellow 44), for vivid lemon shades, a mid tone, e.g. Carbolan Yellow 4G (C.I. Acid Yellow 70), Xylene Milling Yellow 3GL (C.I. Acid Yellow 75), or Cibalan Brilliant Yellow 3GL (Ciba), for less green yellows, and a redder yellow, e.g. Carbolan Yellow R (C.I. Acid Yellow 71), for even redder hues. Orange and brick-red dyeings are produced with a homogeneous dye, e.g. Polar Orange R (C.I. Acid Orange 45) or Coomassie Fast Scarlet 2G (C.I. Acid Red 140), rather than by mixing a red and a yellow dye. For reds a very wide range is available, there being almost every imaginable shade of homogeneous dyes from Coomassie Fast Scarlet 2G (ICI) to Polar Brilliant Red 10B (C.I. Acid Violet 54). It is quite common for a single dyehouse to use six or even more different red fast acid dyes to produce the normal range of hosiery reds. This applies also to blue dyes, where there are all hues from the recently introduced greenish-blue Carbolan Brilliant Blue 2G (ICI) to the reddish Brilliant Alizarine Milling Blue 2RL (C.I. Acid Blue 126) and indeed the Brilliant Alizarine Milling Violet FBL (C.I. Acid Violet 48), each member being used according to the shade required.

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Reference will be made later to advances in methods of applying fast acid dyes, but the choice of dye for any given colour is naturally governed not only by the particular shade but also to a lesser extent by the dyeing process to be employed. At this stage, it may be said that to obtain good levelness it is generally advisable to avoid using dyes which exhaust rapidly, and particularly those whose exhaustion cannot be retarded. Fortunately, there are relatively few milling acid dyes to which this applies, and in all cases there are suitable alternatives of adequate fastness. This does not, however, apply to the chrome dyes.

(b) *Chrome Dyes*

As the lakes formed between chromium and mordant dyes are with very few exceptions relatively dull, the chrome dyes cannot be used for bright colours. They are, however, of the greatest importance for the widely used group of dark, heavy dyeings, such as maroon, navy, bottle green, brown, and black, since not only do they give very good fastness to washing (usually at least up to the standard of the S.D.C. No. 3 Washing Test), but also they have fairly good level-dyeing properties. Furthermore, their cost is less than that of the fast acid or metal-complex dyes which would otherwise have to be used for these colours, and indeed in many cases less than that of level-dyeing acid dyes.

TABLE II

Date of Introduction of certain Milling Acid Dyes

Dye	*Colour Index* No.	J.S.D.C. Reference
Polar Brilliant Red 10B	C.I. Acid Violet 54	50, 391 (1934)
Brilliant Alizarine Milling Blue BL	C.I. Acid Blue 80	53, 442 (1937)
Carbolan Yellow 3G	C.I. Acid Yellow 72	53, 440 (1937)
Carbolan Green G	C.I. Acid Green 27	54, 484 (1938)
Polar Brilliant Red BN	C.I. Acid Red 130	55, 38 (1939)
Carbolan Brilliant Green 5G	C.I. Acid Green 28	60, 288 (1944)
Polar Red RL	C.I. Acid Red 174	62, 59 (1946)
Carbolan Brilliant Blue 2R	C.I. Acid Blue 140	63, 154 (1947)
Carbolan Fawn R	C.I. Acid Brown 49	63, 154 (1947)
Coomassie Ultra Sky SE	C.I. Acid Blue 112	63, 241 (1947)
Brilliant Alizarine Milling Blue 2RL	C.I. Acid Blue 126	63, 241 (1947)
Carbolan Yellow R	C.I. Acid Yellow 71	63, 329 (1947)
Carbolan Yellow 4G	C.I. Acid Yellow 70	63, 329 (1947)
Carbolan Crimson 3B	C.I. Acid Red 139	63, 329 (1947)
Xylene Fast Bordeaux P	—	66, 643 (1950)
Cloth Fast Brilliant Red 3BW	—	68, 516 (1952)
Brilliant Alizarine Milling Blue FGL	C.I. Acid Blue 143	68, 518 (1952)
Brilliant Alizarine Milling Red FBL	C.I. Acid Red 143	69, 451 (1953)
Brilliant Alizarine Milling Violet FBL	C.I. Acid Violet 48	69, 451 (1953)

The above dyes are generally taken very much for granted, and it is seldom remembered that in the First Edition of the *Colour Index* (1924) there were very few milling acid dyes of the types used today. With the exception of Polar Red G (C.I. Acid Red 85) (C.I. 430), Wool Fast Blue BL (C.I. Acid Blue 59) and Wool Fast Blue GL (C.I. Acid Blue 102) (C.I. 833), Alizarine Cyanine Green G (C.I. Acid Green 25) (C.I. 1078), and the Sulphon Cyanines (C.I. Acid Blues 113, 116, and 120) (C.I. 288 and 289), practically none of the bright yellow, blue, red, violet, and green dyes now used is mentioned. Table II gives the year in which reference was first made in the

The large-scale use of chrome dyes on wool yarn is of relatively recent introduction. During the last two or three years, papers by Rattee [11] and by the present author [12] have drawn attention to the advantages to be gained by using the good migrating properties of many of the older after-chrome dyes and then chroming them under suitable conditions so as to produce maximum wet fastness. In contrast to the recent discovery of so many of the important milling acid dyes, most of the chrome dyes used on yarn are at least 50

years old, and the later chrome dyes, most of them possessing good affinity from a metachrome liquor, are used more for loose wool and slubbing than on yarn.

It was mentioned that a considerable number of different milling acid dyes are used for obtaining the bright colours, and that there are very large gaps in the range of dyes of this type. In the case of the chrome dyes, a relatively small number of dyes are used, mainly because only a few chrome dyes are suitable for yarn dyeing, and unfortunately one or two chrome dyes which are very useful on loose wool and slubbing cannot be used on yarn because of inadequate level-dyeing properties. Particular examples are Chrome Fast Yellow G (C.I. Mordant Yellow 16), which is a useful basis for rich browns, and, even more important, Alizarine Blue Black B (C.I. Mordant Black 13), so widely used for dulling other chrome dyes, and Eriochrome Azurol B (C.I. Mordant Blue 1), which is invaluable for imparting bloom to navy blues. The various bright chrome violets, whilst not quite so difficult to dye level as is Eriochrome Azurol B, leave very little margin for safety, and as the navy blues used in the hosiery trade are generally brighter than those on woven fabrics, the absence of bright chrome blues and violets of good levelling properties is particularly serious. None of the alternatives is altogether satisfactory: e.g. the Wool Fast Blues BL and GL, whilst possessing richness of colour, leave much to be desired in wet fastness in medium and full depths, and are not fast enough to light in paler depths.

Whilst most of the newer chrome dyes are specifically designed for application by the metachrome process, mention may be made of two recent afterchrome dyes whose shades are of interest to hosiery-yarn dyers. These are Chromoxane Brilliant Red BL (C.I. Mordant Red 27) (1939) and Eriochrome Brilliant Green GL (C.I. Mordant Green 5) (1947). The former is particularly bright in colour, but is somewhat troublesome because of the way in which it forms a highly resistant coating on stainless steel during dyeing. An interesting feature of Eriochrome Brilliant Green GL is the pronounced change in hue (from red to green) which takes place on chroming.

Dyeing with afterchrome dyes is probably more tricky than applying the other types of fast-to-washing dyes, and there has for some years been controversy regarding the relative merits of the afterchrome and metachrome processes. Mention will be made later of problems in applying afterchrome dyes, but there is little published work on yarn dyeing by the metachrome process. The outstanding fastness of the newer metachrome dyes to light and wet processes is well recognised, but as far as yarn dyeing is concerned there is much to be said for the use of the neutral-dyeing metal-complex dyes for the dull pale and medium depths.

(c) *Metal-complex Dyes*

The first metal-complex dyes, the Neolans, date from 1915 [7], and have been used to a considerable extent on yarn because of their combination of good migrating properties and good fastness to light and wet treatment. They have probably been more widely used on the coarser wool qualities then on fine botany hosiery yarn, where there is some suspicion that the strongly acid dyebath conditions impair the handle of the yarn. The newer neutral-dyeing metal-complex dyes, the first of which (Polar Grey BL, now Irgalan Grey BL— C.I. Acid Black 58) became available in 1949 and new examples of which are still appearing with improved properties each year, have a number of advantages, particularly as regards fastness properties, while the dyebath conditions do not impair the handle of the wool. These dyes are naturally dull, but they are very useful for the mode shades, e.g. silver, beige, etc. They are much simpler to apply than are chrome dyes, and it is expected that they will become much more important as fastness requirements become more stringent and as confidence in applying them to yarn increases.

(d) *Unsolved Problems*

In spite of the large number of fast-to-washing dyes of the milling acid, chrome, and metal-complex types available, there are still a number of requirements which are difficult to fulfil. Some of the more serious of these problems are—

(i) Light fastness
(ii) Accurate reproduction of colour
(iii) Heat sensitivity
(iv) Solidity of shade
(v) Adequate fastness to washing under extreme conditions
(vi) Staining of cotton and viscose rayon during washing
(vii) Artificial-light tone
(viii) Stability during storage.

(i) *Light Fastness*

The most important examples of the very bright popular colours for both machine-knitting and hand-knitting yarns are peach and pink shades, which can be produced only with dyes of the type of Sulpho Rhodamines B and G (C.I. Acid Reds 52 and 50). Whilst their poor light fastness is a constant source of embarrassment to the producers of such dyeings, it is impossible to persuade the public to accept a colour which, whilst not quite so bright, would have adequate light fastness. Vivid yellows dyed with Brilliant Sulpho Flavine FF (C.I. Acid Yellow 7) are in the same category, but have never been as popular as the peaches and pinks which are used in babywear and underwear. Other colours, e.g. bright oranges or bright greens, which have to incorporate a proportion of these fluorescent dyes are also unsatisfactory in light fastness, since the fugitive component fades out quickly, and even though 80% of the total depth may have been obtained with dyes of good light fastness, the net effect is that of the fugitive component. A bright orange dyed with Xylene Light Yellow 2G (C.I. Acid Yellow 17) and Sulpho Rhodamine G (C.I. Acid Red 50) turns to a yellow after only a few hours' exposure to sunlight.

Until the last few months, it would have been necessary to include also bright greens and bright turquoises as being impossible to produce with a light fastness better than 2–3 on the *B.S.* 1006 scale. It has now been found possible to apply direct dyes of the phthalocyanine type with sufficient freedom from skitteriness, even though the degree of levelness is not such as enables them to be dyed on yarn. Experience with dyeings of this type used for outerwear during the summers of 1956 and 1957 has shown that they possess a perfectly satisfactory level of fastness to light and washing. Similar colours based on Patent Blue A (C.I. Acid Blues 5 and 7) and Kiton Green V (C.I. Acid Green 16) would certainly have given rise to an objectionable degree of fading.

Other bright dyeings often appear to be on the borderline of acceptability, and any new bright dyes which will enable a minimum light fastness of 4 to be produced are obviously of the greatest interest to the producers of hosiery yarns. Conversely, any new colouring matter, no matter how bright or how fast to washing it may be, is certain to give rise to complaints from the ultimate user of the garment unless it has a fastness to light of at least 3. An example of this is seen in the introduction of the various fluorescent brightening agents during the past seven or eight years. Wool which has been peroxide-bleached has some tendency to become yellow if exposed to direct sunlight while in a wet alkaline condition. This tendency is greatly accentuated by the application of these fluorescent brightening agents. Graham and Statham [13] recently showed that discoloration is independent of the type of fluorescent brightening agent, and one is led to wonder whether it will ever be possible to produce these dazzling whites on wool with a tolerable level of light fastness. It may be argued that deliberately to introduce products of inferior light fastness is a retrograde step. Certainly, no firm valuing its reputation views such products with favour, and they are used only out of sheer necessity.

(ii) Accurate Reproduction of Colour

In general, as the washing fastness of a dye increases, its level-dyeing properties decrease. This raises difficulties when it is necessary to make additions of shading dyes during yarn dyeing. With acid dyes it is common practice to add shading dye at the boil without in any way altering the dyebath conditions. The uptake of the dye is rapid, but migration enables a level result to be obtained in 30 min. or even less. With most of the fast acid dyes any attempt to add to a boiling liquor would result in almost instantaneous uptake of the dye and hence unlevelness. It is the writer's opinion that addition of shading dye, certainly with fast dyes and also with many dyes generally regarded as being level-dyeing, but actually possessing either high rate of strike or only moderate migrating properties, e.g. Alizarine Supra Sky R (C.I. Acid Blue 67) and Amido Naphthol Red 6B (C.I. Acid Violet 7), is one of the most prolific causes of unlevelness in yarn dyeing. With fast dyes, therefore, some compromise must be reached, either by using dyes possessing a relatively low rate of exhaustion from a boiling liquor and hence usually of somewhat inferior washing fastness, or by cooling the liquor in some way. If much shading dye has to be added, the former course leads to a reduction in washing fastness, while the cooling of the dye liquor involves running-off part of the liquor and making up with cold water. This in turn means a complete alteration in the equilibrium conditions of the dye liquor, with a danger of desorption of part of the dye already present on the wool. In the case of chrome dyes, addition of shading dye is even more complicated, since by the time the dyer has been able to examine a sample, the yarn has been fully chromed. Consequently, any additions of chrome dye are in effect made on wool which has been chrome-mordanted, and in addition the liquor is usually fairly strongly acid. This gives rise to the likelihood of too rapid absorption of the added dye, and in such cases it is certainly preferable to shade with a dye other than of the mordant type. Adjustment of the colour by adding dye to the liquor is thus a most critical procedure, and as a carpet yarn probably receives a larger number of additions of shading dye than other types of yarn, there is obviously an even greater danger of unlevelness due to over-rapid exhaustion of the added dye than with other types of yarn.

(iii) Heat Sensitivity

A number of the fast-to-washing dyes in general use, particularly the chrome reds, the chrome browns, and navies of the Sulphon Cyanine type, are very sensitive to temperature and to moisture content. A particularly serious example is Eriochrome Red B (C.I. Mordant Red 7), which when taken from the sample dryer is much bluer than when the same dyeing has been allowed to condition for 48 hr. Navy blues dyed with Sulphon Cyanines behave similarly, the dyeing becoming much bluer and brighter when it has cooled for a few hours after drying. During this period there is a gradual alteration in hue, and it is very difficult to know how much allowance to make for the "cooling-off" of these heat-sensitive dyeings. Many attempts have been made to overcome the problem, e.g. by matching a wet sample from the dye-vat against a wet standard, wetting out both standard and sample in ether or a similar solvent which cools both patterns, but no really adequate solution to this vexatious problem has been found, and the only answer at present lies in the skill of the individual dyer.

(iv) Solidity of Shade

There are a number of references in the literature to the problems of obtaining solid shades on "tippy" wool, i.e. wool which has been damaged by weathering of the tips while the wool was on the sheep. Townend and Simpson [14] have referred to this matter in the case of acid dyes, and the series of papers by Race, Rowe, Speakman, and Vickerstaff [15] discusses the problem in connection with the faster dyes. Another aspect of the same problem is that of obtaining uniformity of colour in yarn or fabric produced from wool of different qualities. In such cases, the difference in dyeing

properties between the individual fibres can be considerably greater than that between the roots and the tips mentioned above, and this is probably a more serious cause of skittery dyeing than the different behaviour of roots and tips of individual fibres. The problem is usually more severe with coarse wool qualities than with finer wools, and is particularly troublesome with carpet wool. In general, the level-dyeing acid dyes are very good for covering such variations in dyeing properties. It is possible to obtain skittery results with such dyes, but only by deliberately adding a resist agent, such as the Taninol WR (ICI) mentioned by Townend and Simpson[14], and even then the normal combination of red, yellow, and blue acid dyes does not give a great contrast.

When dyeing with fast-to-washing dyes, however, there are certain very important colours which can be produced only with dyes possessing great fibre selectivity under normal dyebath conditions. An outstanding example of this is Brilliant Indocyanine G (C.I. Acid Blue 90), which has for so long been brighter than any other dye of similar tone and which has a great tendency to selective dyeing even when applied along with auxiliary products designed to minimise skitteriness. This shows up to a particularly objectionable degree in bright greens, when the yellow component tends to dye uniformly and the blue component dyes only certain fibres, or even only parts of certain fibres. The recently introduced ranges of reactive dyes of the types of the Procions (ICI) and the Cibacrons (Ciba) are even worse in this respect. It is unfortunate that there is at present no known method of obtaining commercially level results at all depths with these dyes, and it is to be hoped that development work will lead to an adequate solution of this problem.

(v) *Adequate Fastness to Washing under Extreme Conditions*

With the exception of the bright pinks and peaches which can be produced only with dyes of the Sulpho Rhodamine type, and also certain bright yellows and oranges, which have to be dyed with Brilliant Sulpho Flavine FF (C.I. Acid Yellow 7) and the Sulpho Rhodamines, there are now no colours which cannot be obtained fast to the S.D.C. No. 2 Washing Test. This problem of the bright fluorescent colours is now so well known as to need no further emphasis. There are, however, problems arising from the use of fast dyes which from time to time give rise to difficulty. The S.D.C. No. 2 Washing Test as normally carried out uses scoured, undyed white wool and white cotton to assess the degree of staining. In practice, however, a knitted garment containing white wool would never be produced from scoured-only yarn and the white would always be peroxide-bleached or, more commonly, peroxide-bleached and sulphur-stoved. These processes appear to damage the surface structure of the fibre, thereby making it more accessible to any very slight amount of loose dye removed from the dyed material. Consequently, although a dyeing may stand up without difficulty to a conventional washing test, the possibility arises of serious bleeding taking place during domestic washing, and in fact it is difficult to dye many of the heavy reds, royal blues, and bottle greens in such a way that bleeding into a bleached white will not occur, particularly if the proportion of white wool to coloured wool is low.

During the past few years there has been a large increase in the use on the domestic market of washing powders containing sodium perborate. On the whole, these powders, even though many of them give somewhat more alkaline solutions than the soap flakes hitherto generally used for the hand-washing of knitted garments, have not given any particular trouble when attention is paid to the washing instructions issued by the soap or detergent manufacturers, and difficulties usually arise only in cases of gross misuse. A somewhat unexpected problem, however, has manifested itself with Brilliant Indocyanine 6B (C.I. Acid Blue 83). This dye is usually considered to be brighter than any other reddish-blue acid dye; its light fastness, although not outstanding (a medium depth of this dye is the No. 3 Standard on the *B.S.* 1006 scale), rarely gives rise to fading complaints; and its washing fastness in solutions containing no oxidising agent is very good. On washing at temperatures exceeding 60°c., which is, incidentally, much hotter than that recommended by the makers of the washing powders or by the producers of the garments, this dye is oxidised to a grass green, which cannot be restored to royal blue. It has been found necessary by several firms of dyers to discontinue the use of this dye, but any alternative recipe which is fast to sodium perborate lacks the brilliance of Brilliant Indocyanine 6B, particularly in artificial light, and it is unfortunate that this step has had to be taken merely because of the lack of care in washing wool garments. No other dye in common use is decomposed in this way, but naturally any colour produced with level-dyeing acid dyes would be almost completely stripped by the hot, highly alkaline solution which brings about the decomposition of Brilliant Indocyanine 6B.

Although many of the recently introduced detergent and soap powders contain fluorescent brightening agents, it is a matter of some relief to firms engaged in the dyeing of wool that these agents have very little affinity for wool under domestic washing conditions, and that consequently the complications experienced by the cotton and rayon trades have not been shared so far by the wool trade.

(vi) *Staining of Cellulosic Fibres during Washing and during Perspiration*

Many of the important milling acid dyes, particularly those of the azo type, are very closely related to direct cotton dyes. Serious staining of cellulosic fibres occurs in union dyeing, but this by itself is of no significance in the dyeing of all-wool yarns. However, it is possible for an undesirable amount of staining of cotton or rayon underclothes to take place, particularly during wear by a person who perspires freely. Such stains are often not removed from the cellulosic fibre by domestic washing,

and the only method of removing them is a reduction–clear. There is at present no means of overcoming this defect, since there are no alternative dyes possessing adequate fastness to washing on the wool which do not stain the cotton in this way.

(*vii*) *Artificial-light Tone*

In changing over recipes for any given colour from acid dyes to fast dyes, each with different reflectance characteristics, it is not uncommon to encounter a serious difference in the appearance of the two dyeings when viewed under different types of illumination. These difficulties, which have existed for many years, have been complicated by the rapid introduction of the various types of fluorescent lighting in addition to incandescent electric lighting and the many forms of daylight. Similar problems arise also when standard and pattern are examined by persons whose colour-viewing characteristics are different. Considerable difficulties arose from this in the early days of the neutral-dyeing metal-complex dyes, but the introduction of such dyes as those metal-complex browns which become redder in artificial light has done much to minimise the difficulty. Nevertheless, the problem is still present with all forms of fast dye, and much ingenuity is often required to obtain a workable solution.

(*viii*) *Stability during Storage*

Deterioration during storage, due to the effect of sulphur dioxide on certain azo dyes based on G acid, particularly Polar Red G (C.I. Acid Red 85) and Orange GG (C.I. Acid Orange 10), is well known. The tendency of such dyes to form azo-sulphite compounds usually much yellower and weaker in colour than the original was described by Goodall [16]. Many agencies have been found to be responsible for liberating these traces of sulphur dioxide, e.g. sulphite wrapping paper, fumes from coke and gas fires, storage near yarn treated with sulphur dioxide, etc. Unfortunately, there is no real solution to the problem other than to discontinue using such dyes, since the change in colour can occur slowly and insidiously many months after the material has been dyed. Fortunately, alternative dyes of at least equal fastness to light and washing and of good stability to sulphur dioxide are available.

From time to time a similar change of colour has been observed in dyeings produced with chrome browns of various types. Nearly all the commonly used chrome browns have poor fastness to sulphur dioxide. The change of colour is much less pronounced than with the azosulphite-forming dyes mentioned above, and probably arises from the change in pH of the wool due to combination with sulphur dioxide and the resulting formation of sulphuric acid. Most of the brown chrome dyes contain one or more free amino groups in the molecule, and presumably combination with acid leads to conversion of these amino groups into ammonium groups, with a corresponding change in colour.

3. DYEING METHODS

In spite of advances in the design of dyeing machines and in the introduction of new dyes, much of the success in obtaining level dyeings with fast-to-washing dyes has come from improved techniques in applying these dyes. It must be admitted that much of the experimental work has been carried out on an *ad hoc* basis, and there is little in the literature to guide anyone working in this field.

Experience shows that the best results in dyeing yarn with any type of fast dye are given by ensuring that absorption is slow and uniform. Any agent which may lead to sudden uptake of dye at any stage of the dyeing process must be avoided, and for this reason the standard techniques which are successfully employed in dyeing loose wool and slubbing, where it is the general custom to add acid at one or more stages during the course of dyeing so as to obtain good exhaustion, are highly dangerous on yarn. The reason for this is that in all existing designs of dyeing machine, even with the best possible circulation of the liquor, it takes some 2–3 min. for the dye liquor to penetrate completely through the yarn. Thus, some parts of the material come into contact with the liquor a considerable time before other parts. If acid is added to the hot liquor, it will combine rapidly with the outer surfaces of the load, and exhaust itself on to these parts, leaving the centre practically unchanged. As the dye remaining in the liquor reacts more rapidly with wool containing acid than with wool having a lower acid content, the dye will in effect follow the unevenly absorbed acid and give an uneven dyeing. Because of the absence of any migration with most forms of fast dye, unlevelness arising in this way is impossible to correct. Mention has been made previously of the similar type of trouble which can arise from injudicious addition of shading dye, which again tends to be taken up at the parts of wool with which the dye liquor first comes in contact. These difficulties arise with all forms of fast yarn-dyeing, but the methods employed for the individual classes will now be considered.

(*a*) *Fast Acid Dyes*

Much more attention has been paid by academic workers to the level-dyeing acid dyes than to the fast acid dyes. This may be seen from the book by Vickerstaff [17], where reference to the fast acid dyes is very brief by comparison with the space devoted to the level-dyeing acid dyes, and particularly that devoted to the dyes used for cellulosic fibres. This neglect is probably due to the relatively recent introduction of many of the more important members of the milling acid group, but systematic research on this subject would lead to valuable results, particularly as the mechanism of dyeing appears to differ very greatly from that with other types of wool dye. An introduction to the theory of the dyeing of fast acid dyes was made by Goodall [18], who drew attention to the colloidal nature of these dyes, particularly at temperatures below the boil. In a later paper by Goodall and Hobday [19] it was shown that the milling acid dye Polar Yellow R (C.I. Acid Orange 63) had an acid-combining capacity of the order of 140 ml. of N. dye acid per 100 g. of wool at 100°C. and pH 1·4, as compared with the theoretical figure of about

80 ml. This was believed to be due to sorption in the form of aggregated particles. Most published methods for applying dyes of this class refer to dyeing from an acid liquor; e.g. Vickerstaff[17] speaks of applying milling dyes at pH 4–6 and super-milling dyes at pH 6–7. Whilst applying these acid milling dyes in presence of acid will certainly bring about exhaustion, other factors must be considered when it is desired to obtain not only a reasonable degree of exhaustion but also good levelness. From a practical point of view, there are two general methods for dyeing with milling acid dyes—(i) pH control and (ii) electrolyte control. It is, of course, common practice to use both methods simultaneously, and in both cases great attention has to be paid to the particular sensitivity of these dyes to temperature.

(i) *pH Control*

Lister[20] has referred to the wide variations which exist between different types of dyes in the equilibrium pH required to bring about 90% exhaustion of the dye liquor. Whilst it has been found in practice that 90% exhaustion is probably somewhat too high for safety, and that a figure of 80% would represent a better compromise between ensuring level dyeing and achieving reasonable economy from the dye, his findings work out very well for the application of milling acid dyes to wool yarn. It is evident that very close control of the pH is essential to avoid too rapid exhaustion, which is, of course, much more serious than too slow exhaustion. In the discussion on his paper, Lister stated that it appears desirable that components of mixtures should have their optimum pH values for dyeing within $\pm 0\cdot2$ unit. This is frequently impossible, since, in matching given colours, the tone of the pattern dictates the selection of the dyes, and it is then necessary to select dyes which are not completely compatible from a dyeing point of view. In such cases the only possibility when dyeing with pH control is to dye at the pH value required for the component of greatest acid sensitivity.

Reference has already been made to the dangers of adding acid during dyeing. It would appear from practical experience that this applies no matter whether the acid is added quickly, is entered by means of a drip-feed, or arises from liberation from other substances. Trials with the use of hydrolysable esters by the method of Hannay and Major[21] have shown that the liberation of acid from such compounds during dyeing can lead to unlevelness.

For yarn dyeing with fast acid dyes, especially those of the highest wet fastness, the most widely used methods are based on ammonium salts, usually the sulphate, phosphate, or acetate. These do not give the high degree of exhaustion which would be required in a slubbing dyehouse, but on the whole it is possible to produce adequate levelness and the necessary fastness to wet treatment. Contrary to widely held beliefs, ammonium salts do not appear to liberate ammonia during the course of dyeing, and the apparent formation of acid during dyeing is evidently due to changes in dissociation of the ammonium and acid ions as the temperature of the dyebath increases. Peryman[22] has published some most interesting results on the changes in the pH of solutions of ammonium salts in the concentrations normally found in dye liquors. These show that ammonium sulphate solutions, although apparently at pH 6·37 at room temperature, can have a pH as low as 4·9 at the boil. Ammonium acetate, on the other hand, whilst apparently at pH 7·38 at room temperature, falls to only pH 6·2 at the boil, while the corresponding change for a mixture of diammonium phosphate and ammonia is from 8·48 at 18°C. to 7·2 at the boil. This presumably explains why ammonium sulphate invariably gives much better exhaustion, but with a greater risk of unlevelness, than ammonium acetate, while the exhaustion from dyebaths containing mono- or di-ammonium phosphate often appears disappointingly low.

Within recent months, there has been considerable interest in dyeing with fast acid dyes at relatively low pH values (ca. 4) by forming complexes with auxiliary products, the exact nature of which has not been disclosed, but which are probably either cationic or polyethenoxy compounds of pseudo-cationic nature. With fast acid dyes these compounds form aggregates which break down at high temperatures, thereby liberating dye at a regular slow rate. The mode of action can be considered as competition with the fibre for the dye, this being the reverse of the generally accepted action of sulphate ions in dyeing wool with level-dyeing acid dyes, where the sulphate ion competes with the dye for available sites on the fibre. It is believed that some of these auxiliary products contain, not only an aggregating agent, but also a dispersing agent to break up the aggregate and prevent precipitation. Work on these compounds is still under development, and doubtless improved products will appear, since obviously a reliable product would be of great assistance, not only as a levelling agent, but also as a partial stripping agent which could be employed for correcting unlevel or off-shade batches. It must be pointed out that experience so far indicates that dyes react individually to these products, and in a combination of three dyes one may be virtually unaffected, another be slowed down in its rate of absorption, and a third almost completely restrained. Another difficulty which has been observed in practice is that certain of these retarding agents have a marked affinity for wool and have a tendency to be absorbed unevenly, thereby promoting unlevelness, and in view of this high affinity for the wool, corrective treatment may be very difficult.

The mode of action of sodium sulphate when it is used in combination with acids or acid-yielding salts in the dyeing of fast acid dyes is entirely different from that when it is used in conjunction with sulphuric acid for applying level-dyeing acid dyes. With the latter the sulphate ion has been proved to reduce exhaustion and promote migration. With the fast acid dyes the reverse is the case, and in general this is more noticeable as the wet fastness of the dye increases. It should be emphasised that with wool dyes other than those

of the level-dyeing type, Glauber's salt certainly does not improve either level dyeing or migration. It is in fact possible to use sodium sulphate as an exhausting agent, and this method will now be discussed.

(ii) Electrolyte Control

This method of applying fast acid dyes is generally similar to the dyeing of cellulosic fibres with direct dyes. Lister[20] observed that: "dyes with known unlevel dyeing properties were more sensitive to the salt addition than the level dyeing type", and Vickerstaff[17] gives details of some unpublished work by Meggy and Lemin. The latter workers showed that, whereas Coomassie Fast Scarlet B (C.I. Acid Red 111), which is widely used for producing scarlets of good washing fastness, exhausts to only 22% in absence of electrolyte, when dyed in presence of 30% sodium chloride on the weight of wool the exhaustion rises to 83%. A considerable number of other dyes representing a full range of hues behave similarly, and in this way it is possible to dye practically all bright colours by "feeding-on" the dye with sodium sulphate or, less commonly, sodium chloride. The technique employed is usually to dye at a pH value of 6 or above, so that the likelihood of salt formation between fibre and dye is negligible. It is in fact not difficult to apply many fast acid dyes at pH values of over 8, but the reasons why this is not generally done will be mentioned later. Auxiliary products, usually sulphonic acids of naphthalene derivatives, are frequently found beneficial when dyeing by this technique. Whether they act by competing with the dye for places on the wool fibre in a similar manner to the sulphate ion in dyeing with level-dyeing acid dyes, or whether they act as dispersing agents for dyes which have become more or less aggregated by the relatively high electrolyte concentration, is uncertain, as there is very little knowledge of the mechanism of dyeing by such processes.

When dyeing with fast acid dyes, no matter what method is adopted, certain complications must be guarded against—

(A) Wool contains a yellow pigment as a natural ingredient, the amount generally being greater on the coarser wools than the finer botany qualities. This is particularly the case with the coarse wools used in carpets, where it is often found necessary to use selected blends of good whiteness for pale colours. Even under acid dyeing conditions, this yellow pigment raises serious difficulties with many wool qualities, since in pale colours the yellow pigment contributes an appreciable proportion of the colour seen by the eye. It has poor light fastness, often being little better than the No. 1 Standard on the *B.S.* 1006 scale. As the pH of the dye liquor increases, the tendency of the wool to become yellow during dyeing increases. This makes it difficult to produce bright pale colours unless the wool is subsequently peroxide-bleached. For the duller colours such as silver and beige, bleaching is not regarded as necessary, but even though dyes known to have a high light fastness may be used, the actual colour produced can often exhibit an undesirable degree of fading purely due to the effect of light on this yellow pigment.

(B) Peryman[23] referred to the effect of the pH of the dye liquor on the disulphide content of wool. Whereas at pH 6 about 90% of the disulphide sulphur remains unchanged, at pH 8 only 60% remains after boiling for 3 hr. Schöberl and Wagner[24] have recently summarised their work on the mechanism of the conversion of cystine into lanthionine, and it is likely that this is the change which takes place during dyeing at pH values above 6. The presence of hydrogen sulphide can easily be detected near yarn dyed at pH 8 or over, and presumably arises from attack on disulphide bonds. If no corrective measures are taken, such a pH can readily occur from carryover of alkali from the scouring, or from the conversion of carbonate into bicarbonate if base-exchange-softened water is used. The formation of lanthionine in itself does not necessarily result in any noticeable deterioration of handle, tensile strength, or colour, but it renders the wool almost impervious to subsequent shrink-resist treatment. In view of the growing importance of the latter, such a change in the chemical behaviour of the wool is a serious consideration.

(b) Chrome Dyes

During the past few years some attention has been given to the theory of the dyeing of wool with chrome dyes, and papers specifically relating to the level-dyeing properties of the chrome dyes have appeared by the present writer[12] and by Rattee[11]. In general, it appears that dyes of low molecular weight have good migrating properties and can be dyed level, at any rate until they have been chromed. As with fast acid dyes, care must be taken to select the correct initial pH so that no addition of acid has to be made during dyeing. The behaviour of the dyes during chroming is very critical, and particular attention has to be paid to this stage of the process. Just as no acid is added for exhaustion during dyeing, it is equally fatal to add acid along with the chrome. For various reasons there is much to be said for cooling the liquor between dyeing and chroming so as to start the latter process at a temperature of under 90°C. Given correct selection of dyes— and there are a number of suitable yellow, orange, red, violet, navy blue, green, and black chrome dyes available — the application of the dye prior to chroming is little more difficult than that of level-dyeing acid dyes. The following complications can, however, arise during chroming—

(i) With dyes of low affinity, e.g. certain chrome yellows, there is a danger of partial desorption if too much of the dye liquor is replaced by cold water for cooling prior to chroming.

(ii) Complete destruction of the dye can occur during chroming. This is, perhaps, not so serious as in the chroming of dyes on nylon, where Hadfield and Sharing[25] have shown that it is essential for some reducing agent to be added to the chroming liquor. Wool acts as its own reducing agent, but even so, chroming conditions which are too drastic, e.g. the presence of too much chrome or too low a

pH, can certainly decompose a few extremely useful dyes, e.g. Eriochrome Red B (C.I. Mordant Red 7). Furthermore, variations in chroming conditions between successive batches are certain to give rise to lack of continuity of colour, which is very troublesome and time-consuming, since it necessitates an excessive number of additions of shading dye. It has already been mentioned that certain chrome dyes present difficulties because of their change in colour when the dyeing is cooled, but the changes arising from slight alterations in dyeing technique are even greater.

(iii) Attempts to chrome too rapidly can lead to uneven chroming. This can give rise to local destruction of dye, but other dyes, e.g. certain of the chrome browns, can be oxidised to give increased tinctorial yield. In either case the colour becomes unlevel and is very difficult to control.

(iv) Chroming at too low a pH apparently oxidises the cystine link to sulphoxides in much the same way as do many of the commercial shrink-resist processes. The result is that the wool becomes highly shrink-resistant. For the hosiery trade this is not a disadvantage, since in any case the wool is likely to receive some treatment of this type at a later stage. For yarns to be used in weaving, however, where it may be necessary for the cloth to be milled at a later stage, this can be a serious handicap: e.g. uneven shrinkage of sections of the warp or weft can give rise to most objectionable patterning.

(c) *Neutral-dyeing Metal-complex Dyes*

These dyes, which first became available about 1949 and members of which are still regularly appearing, are at present receiving a great deal of examination and are finding acceptance for pale and medium depths of high fastness. Before the introduction of these dyes, these depths had to be obtained on yarn with the small number of grey and brown acid milling dyes, e.g. Alizarine Light Grey BBL (C.I. Acid Black 48) or Alizarine Light Brown BL (C.I. Acid Brown 36), while on loose wool and slubbing the fast metachrome dyes were employed.

The dyeing properties of these neutral-dyeing metal-complex dyes are very interesting and differ in many respects from those of the conventional fast acid dyes. Schetty[26] has given details of their excellent ability to give solid shades on wool of different dyeing properties, and Hirsbrunner[27] recently described their surprisingly good migration properties. These dyes are known to contain no sulpho groups, and they apparently have a feeble negative charge when in solution, which no doubt accounts for the need to use a little acid when dyeing with these dyes. At present, the most widely used methods for applying such dyes employ ammonium salts, but as with the fast acid dyes, attempts are being made to apply them at pH values of 4–5, in presence of an auxiliary product with high affinity for the dye. It is at present too early to say whether this technique will become widely used. It is to be expected that these dyes will become increasingly important, particularly as there are indications that brighter colours will be produced.

* * *

The writer thanks the Directors of Patons & Baldwins Ltd. for permission to publish this paper, and also several colleagues for helpful advice and comments. For information on the history of yarn-dyeing machines, thanks are due to Herr Hans Müller-Busch (Obermaier & Cie.) and Mr. A. Gaunt (T. F. Firth & Sons Ltd.).

PATONS & BALDWINS LTD.
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(*MS. received 19th December 1957*)

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The Editor does not hold himself responsible for opinions expressed by correspondents

The Fixation of Reactive Dyes on Modified Celluloses

In view of the recent publication of an investigation of the dyeing behaviour of a reactive dye on modified celluloses [1] it appears opportune to record the results of some experiments of a similar nature undertaken in an attempt to establish the nature of the fibre–dye linkage.

Daruwalla and Subramaniam [1] have recorded that dichromate–oxalic acid–chlorite oxycelluloses show a greater reduction of dye uptake for a given carboxyl content than do periodate–chlorite oxycelluloses. This they ascribe to the nature of the oxidative attack on cellulose by these reagents. It is doubtful whether dichromate–oxalic acid–chlorite oxycelluloses are equally oxidised in both the crystalline and amorphous regions of the fibre [2], which is the case with periodate–chlorite oxycelluloses. If oxidation is not homogeneous, carboxyl content values could be misleading with regard to the actual relative effects of the two treatments.

The effect on the fixation of two reactive dyes when printed on periodate–chlorous acid and nitrogen dioxide oxycelluloses (both agents being capable of penetrating the crystalline as well as the amorphous regions of the cellulose) has been examined in comparison with their behaviour on the corresponding unmodified cellulose fabric. Nitrogen dioxide [3] causes oxidation mainly at C(6), while periodate [4] oxidation results in attack at the C(2)–C(3) carbon atoms of the cellulose molecule. The dyes examined— Procion Brilliant Red 2B and Procion Brilliant Red H3B (ICI)— were representative of high and low reactivity. They were applied by printing from a paste containing sodium bicarbonate, fixation being by a short steaming treatment [5]. Quantitative measurements of fixation were carried out by the method described for vat dyes [6].

The relative reductions in fixation obtained with Procion Brilliant Red 2B on periodate–chlorite oxycelluloses are in general agreement with those reported by Daruwalla and Subramaniam [1] for dye applied by the cold-dyeing technique. This type of oxidative treatment does not affect the fixation of the less reactive dye to the same extent. At low degrees of oxidation the fixation of Procion Brilliant Red H3B is affected to a lesser extent than that of Procion Brilliant Red 2B; the reverse, however, is the case at higher degrees of oxidation. Similar experiments with nitrogen dioxide oxycelluloses showed that the fixation of Procion Brilliant Red H3B was affected to a greater extent than that of Procion Brilliant Red 2B over the range of degrees of oxidation studied. Table I shows some typical results.

With periodate–chlorite oxycelluloses of higher degrees of oxidation the reversal in the relative effect on fixation of the two dyes may be due to the carboxyl group introduced at C(3) reducing the reactivity of the C(6) hydroxyl group to such an extent that only the more reactive dye can react appreciably.

TABLE I

Type of Oxycellulose	Carboxyl Content (m-moles per 100 g.)	Fixation (%) Procion Brilliant Red 2B	H3B
Control (untreated)	—	61	60
Periodate–chlorite	5·0	43	51
	10·4	27	33
	20·0	21	15
	34·6	18	5
Nitrogen dioxide	10·1	51	46
	16·1	43	31
	20·4	39	25

It should be borne in mind, however, that with both types of oxycellulose two factors are operative in reducing the fixation of the dye— (i) removal of hydroxyl groups, by means of which reaction between dye and fibre can occur; and (ii) introduction of carboxyl groups, which exert a repulsive effect on the anionic dye (cf. the application of direct dyes to hypobromite oxycelluloses [7]). It will be seen from Table I that at an equal carboxyl content a periodate–chlorite oxycellulose gives a lower fixation of dye than does the nitrogen dioxide oxycellulose. This is in contrast to the greater effect shown by the dichromate–oxalic acid–chlorite oxycellulose. It thus appears unwise to draw conclusions as to positions of substitution of reactive dyes in cellulose solely from their fixation behaviour on carboxyl derivatives.

Indirect evidence of the nature of the types of dye–fibre bond present with dyes of differing reactivity was obtained in studies of the periodate oxidation of dyed cotton fabric. Fabric which had been dyed with Procion Brilliant Red 2B and Procion Brilliant Red H3B to the same dye uptake was subjected to oxidation with periodate, when it was observed that the rate of oxidation fell in the order: untreated cotton > cotton dyed with Procion Brilliant Red H3B > cotton dyed with Procion Brilliant Red 2B. These results, together with the fixation results obtained on oxycelluloses, suggest that the more reactive dye reacts to a relatively greater extent with the secondary hydroxyl groups in the cellulose than does the less reactive dye. This is difficult to demonstrate directly, but measurement of the ratio of the first-order rate constants for the reaction of these dyes, under alkaline conditions, with a simple primary alcohol and a simple secondary alcohol (*n*- and *iso*-propanol) showed that the tendency of the less reactive type of dye to react with the secondary alcohol was considerably less than that of the more reactive dye. Thus the ratio—

$$\frac{\text{Rate constant for reaction with } n\text{-propanol}}{\text{Rate constant for reaction with } iso\text{propanol}}$$

for Procion Brilliant Red 2B was 3·7, while that for Procion Brilliant Red H3B was 15·2. Dyes of reactivity intermediate between these two examples gave intermediate values for the reaction rate ratio.

Further evidence for the existence of two types of dye–fibre bond has been obtained from a study of the removal of combined dye from printed fabric by acid hydrolysis. If cotton fabric which has been printed with Procion dye is subjected to the action of hot dilute acetic acid, the dye is gradually removed from the cellulose at a rate dependent upon the particular dye used. This breakdown appears to take place by acid hydrolysis of the ester-like dye–fibre bond. Initially the dye is removed relatively quickly, but after the major portion of the bound dye has been removed, further hydrolysis is relatively slow. The dye which remains more firmly bound to the cellulose appears to correspond to that which has reacted with the secondary hydroxyl groups. This is in agreement with the fact that carboxylic esters of secondary alcohols are more stable to hydrolysis than are the esters of the corresponding primary alcohols. Moreover, if comparison is made of the observed degree of hydrolysis at which further stripping becomes relatively slow and the theoretical value at which this should occur, as calculated from the primary/secondary alcohol reaction rate ratios previously mentioned, there is fairly good agreement between the values obtained (Table II).

TABLE II

Dye	Primary/Secondary Alcohol Reaction Rate Ratio	Dye remaining unstripped (%) Calculated	Dye remaining unstripped (%) Observed
Procion Brilliant Red 2B	3·7	21·2	23·1
Procion Brilliant Blue R	6·6	13·4	15·8
Procion Brilliant Red H3B	15·2	6·2	8·0

In calculating the theoretical figures given above it is assumed that only one of the secondary hydroxyl groups of the cellulose has an appreciable reactivity towards the dye relative to that of the primary hydroxyl. By analogy with reaction rates for the hydroxyl groups at C(6), C(3), and C(2) with *p*-toluenesulphonyl chloride [8–10] it is probable that, apart from the C(6) hydroxyl, it is mainly the hydroxyl group at C(2) which takes part in the reaction with the dye to produce the hydrolysis-resistant bond.

The evidence to date for the position of substitution of reactive dyes in cellulose suggests, therefore, that substitution occurs mainly at C(6), with a minor degree of substitution at C(2). The extent to which the latter process occurs depends upon the particular dye applied.

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Absorption Spectra of Aminoanthraquinones

In three recent papers [1–3] on the absorption spectra of six aminoanthraquinones in relation to their colour and constitution and on their photochemistry, Egerton and Roach make little reference to an extensive survey of the same general nature and covering no fewer than 39 anthraquinonoid vat dyes and related quinones, made by Moran and Stonehill [4–6], beyond an intercomparison of the spectral data for four of the aminoanthraquinones, and two suggestions which are refuted below.

Egerton and Roach [1] suggest that the cause of the disintegration of cellulose thimbles or filter paper observed [4] during solvent extraction or drying of photoactive vat dyes is the presence of sulphuric acid impurity in the dyes. This would be a reasonable explanation were it not for the fact that the disintegration occurred *only* with photoactive dyes, although the inactive dyes had been given the same preliminary acid-treatment.

The second suggestion [2] is that the darkening observed [5] upon light-exposure of cellulose acetate films dyed with 1-aminoanthraquinone is again due to sulphuric acid impurity in the dye. This can scarcely be taken seriously, since Moran and Stonehill dyed their films from a conventional alkaline hydrosulphite vat, as they clearly stated, and not, as in the work of Egerton and Roach, an alcoholic dye solution.

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Notes

Meetings of Council and Committees
July

Council— 2nd

Publications— 15th

Diplomas— 17th

Fastness Tests Co-ordinating— 3rd

Colour Index Editorial Panel— 18th

International Federation 1959 Congress—

Planning and Co-ordinating Committee— 22nd

Social Subcommittee— 16th

Mercer Lecture— 2nd

Manchester College of Science and Technology Chemical Engineering Building

The new extension of the College in Jackson Street was formally opened by Sir Alexander Fleck, K.B.E., F.R.S. (Chairman of Imperial Chemical Industries Ltd.), on 19th May 1958. An old cotton mill has been ingeniously and tastefully converted for use by the Departments of Chemical Engineering and Textile Chemistry. The latter retains the new dyehouse and research laboratories in the main building, and is utilising the new extension mainly for teaching laboratories— for general work and for physical chemistry— each accommodating about 36 students. There are also a research laboratory, an optical room for absorptiometric and colorimetric work, a lecture room, and a small library and museum.

French Light Fastness Testing Station

The "station d'insolation", previously at Aigues-Mortes (cf. J.S.D.C., 72, 354 (1956)), has been transferred to "Les Banlènes", Vauvert (Gard), France. Addressed envelopes for the despatch of patterns may be obtained from the office at 12 rue d'Anjou, Paris VIII.

Discovery of Perlon (Polycaprolactam or 6-Nylon)

A short article by P.-A. Koch in the *Zeitschrift für die gesamte Textilindustrie* (60, 53–54 (5 Jan. 1958)) outlines the discovery of Perlon and gives a brief biography of its discoverer, Dr. Paul Schlack. Interest in the chemical basis of photography led him to study chemistry at the Technische Hochschule in his native town of Stuttgart, after which he undertook protein research in Copenhagen. He was later concerned with modifying the dyeing properties of acetate rayon by means of suitable additions, and in 1929 came to study the amino acids with a view to producing long fibre molecules. The economic crisis stopped these researches, but they were taken up again on publication of the first nylon patents by Carothers. Although the latter had unsuccessfully attempted to polymerise caprolactam in 1930, Schlack heated the hydrochloride of aminocaproic acid overnight with a suitable catalyst, and on 29th January 1938 he obtained the first fibres of Perlon.

The new material was first termed "Perluran", from the works designations of the raw materials, as such a name would arouse less curiosity than a number or a letter. The "lactam process" was patented on 11th June 1938 (*GP* 748,253), and coarse monofils were available in the autumn of 1939. Bulk production of the filament began in 1943 at the new IG works at Landsberg an der Warthe. The lactam process has certain economic and technical advantages compared with 6,6-nylon manufacture, notably in the stability of the melt, and it has therefore spread to many other countries.

Information on Japanese Science

An information centre has been established in Japan, which will provide photocopying and English translation services, available to individuals and organisations outside Japan. The address is— The Japan Information Centre of Science and Technology, C.P.O. Box 1478, Tokyo, Japan.

New Books and Publications

Review of Textile Progress
Volume 8 1956

Manchester: The Textile Institute; Bradford: The Society of Dyers and Colourists. 1957. Pp. 611. Price 42s. 0d. ($6.00).

There can, of course, be no question of the authority and expertise which the contributors bring to the *Review of Textile Progress* each year. Any discussion of the value of the *Review* and any suggestions for modification or for its evolution in any direction must come from an examination of its aims. These have never been formulated in any detail.

There are several sources of information on textile subjects— original research papers in learned journals such as those of the Textile Institute and the Society of Dyers and Colourists, more general review articles and technological research in the trade papers, textile abstracts prepared by the same two societies, the relevant abstracts from less specific organisations like the Society of Chemical Industry and the American Chemical Society, the *Review* under consideration here, and the relevant chapters from the Society of Chemical Industry's yearly *Reports on the Progress of Applied Chemistry*.

Without suggesting that the sources of information available to, for example, the pure chemist are perfect, and acknowledging that they, too, are evolving all the time, I think a comparison of the sources of textile and purely chemical information does show a possible way in which the *Review of Textile Progress* might evolve.

The American Chemical Society's *Chemical Abstracts* correspond to the abstracts published by the Textile Institute and the Society of Dyers and Colourists. The journals of the national chemical societies correspond to the various national textile journals. Significant differences emerge when the Chemical Society's *Annual Reports on the Progress of Chemistry* are compared with what must be their equivalent, the *Review of Textile Progress*: a far larger volume of work is covered in 400 instead of 600 pages; each branch of the subject is not necessarily reviewed each year; and the *Annual Reports* are supplemented by more profound reviews which appear in, for example, *Quarterly Reviews*, published by the Chemical Society, and the more specialised annual reviews such as *Advances in Protein Chemistry*, for which several subjects are chosen for scrutiny every year. The Society of Dyers and Colourists tacitly admits the need for such analytical review articles when it publishes in its *Journal* the invaluable Explanatory Papers on Modern Theory from time to time.

A contributor to the *Review of Textile Progress* has to make an uneasy compromise between a succinct but complete coverage of a year's research and patents, and a critical assessment of some circumscribed part of the subject in which important progress may have been made. The result of this compromise may be seen on almost any page— the paragraphs are essentially abstracts of research papers or patents. Several authors comment explicitly or implicitly on the arbitrariness of one-year intervals between reviews, e.g. the introduction to the chapter on Dyeing (p. 322)—

> Last year's introduction contained the rather wistful complaint that already at the time of writing there were developments to be credited to 1956 which were much more interesting than anything reported during 1955. Faced now with the responsibility of setting these forth . . . .

It would seem best, therefore, if possible, to separate the two functions of the *Review of Textile Progress*— to produce a more compact review of the literature giving details of definite advances in textile science and technology and, in addition, a review, either quarterly or yearly along the lines of *Quarterly Reviews* or *Advances in Protein Chemistry*, which would, like *Quarterly Reviews*, contain "articles by recognised authorities on selected topics . . .".

P. T. Speakman

The Chemistry and Technology of
Naphthalene Compounds

By Norman Donaldson. Pp. xv + 512. London: Edward Arnold (Publishers) Ltd. 1958. Price, 90s. 0d.

This book deserves to succeed if only for the fact that the author has completed a work which at least two others have unsuccessfully attempted before him. During the 'twenties advertisements appeared relating to a forthcoming book on the chemistry of naphthalene by the late Professor Wynne. Rumour has it that, like Lawrence's *Seven Pillars of Wisdom*, the manuscript was lost on a railway station. The book certainly never got into print, and neither did another manuscript which the reviewer was privileged to see in more recent years. Considering the great technical importance of naphthalene compounds, and the fact that naphthalene chemistry is much more subtle and diverse than the relatively simple chemistry of benzene derivatives, it is astonishing that no single-volume work entirely devoted to the topic has appeared since 1888. When one further reflects that naphthalene chemistry is necessarily more cogent to the chemistry of polycyclic aromatic hydrocarbons as a whole, one can see that here was a serious gap in the chemical literature. It is very difficult to see why it has not been bridged before.

Many chemists who have been conscious of this gap must have been looking forward to this new work with keen anticipation, and one can only state with regret that most of them are likely to be far from satisfied with it. In the first place the book is badly titled. It could, perhaps, be more correctly titled "The Technology of Naphthalene Compounds", but even this is not a satisfactory description of its contents. "A Source Book of Naphthalene Chemistry" might be most appropriate. Although the book ranges widely over the field, with special emphasis on compounds of interest to the dye industry, the treatment throughout tends to be encyclopaedic rather than selective and analytical. The author also appears to be not as familiar with modern developments in theoretical aromatic chemistry as one might have expected. This is particularly illustrated in his opening chapter, when he is dealing with the structure of naphthalene. Much work quoted (e.g. the ozonolysis of naphthalene compounds, Fieser's studies on diazo coupling to hydroxynaphthalenes) is no longer given the simple explanations accorded to it here. One can, however, sympathise with the author in so far as even simple problems like the lack of reactivity at the 3-position in β-substituted derivatives are still without satisfactory explanation, and only now is adequate evidence being assembled to assess and interpret internuclear influences on reactivity.

There are, however, very valuable aspects of this work which must not be ignored. A full search of the literature is claimed to the end of 1954 and a partial search to October 1956. Much use has been made of B.I.O.S. and F.I.A.T. reports, this being reflected in the many detailed descriptions of technical processes, which should be of great interest both to manufacturers and to laboratory chemists. There are thirty-one tables (some of two pages or more) and over three thousand references to original literature, and the detailed indexes run to nearly forty pages. It is pleasing to note from these indexes that work in technical colleges in this country in recent years is not to be discounted, somewhat in contrast to the

relatively scant attention that has been paid to naphthalene chemistry in more academic circles. This narrow view of aromatic chemistry, based largely on the reactivity of benzene compounds, is bound to be much altered in the next few years. Intensive studies on the structure and the reactivity of naphthalene compounds are being made by many research schools both in this country and in such widely scattered places abroad as Holland, New Zealand, Australia, and the U.S.A. The results of these investigations will not be of interest to naphthalene chemists alone but are likely to give us entirely new perspectives on aromatic chemistry as a whole.

In the reviewer's opinion this book will be an invaluable addition to most libraries as an up-to-date supplement, and extension, of Volume **12B** of Elsevier's *Chemistry of Carbon Compounds* (which does not deal with patent literature), and individual chemists will want to purchase it as a convenient-sized general reference volume. The binding, layout, and printing of this work are all first class, for which the publishers deserve to be specially congratulated.

E. R. Ward

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Die wichtigsten Farbstoffe, ihre Herstellung und hauptsächlichen Eigenschaften

Compiled from lectures by Professor H. Hopff. Pp. ix + 1–101 and vii + 105–319. Zürich: Verlag der Chemiestudierenden an der Eidgenössischen Technischen Hochschule. 1956. Price, 32.00 Swiss francs.

These volumes are a compilation of notes on intermediates, dyes, and dyeing based on the lectures of Professor H. Hopff. It is pointed out, however, that the publication is not intended as a substitute for these lectures but that it releases the student from the necessity of making basic notes and thus enables him to devote his attention more closely to the lecturer and his subject. Consequently, the salient features of each subject are presented in concise, systematic form; only essential matter is summarised, and it is stressed that no claim to completeness is made for the notes on any subject. Bearing this in mind, the text is printed on one side of the paper only, thereby providing space for the student to insert his own notes supplementing or supplanting the printed notes, adding references to recent work, and, in general, bringing each particular subject up to date. The provision of this facility is particularly useful with a subject which is continually changing and expanding.

Volume I opens with introductory subject-matter and a brief historical outline of the most outstanding discoveries in dyes and dyeing processes following Perkin's discovery of Mauveine. Attention is then directed to the nature and the occurrence of colour and the relationship which exists between colour and constitution; the natural and synthetic fibres; the mechanism of dyeing; and the classification of dyes according to (*a*) chemical structure, (*b*) dyeing properties, together with the fastness properties of dyes and their assessment. A substantial portion of the volume (pp. 28–78) deals with the isolation of raw products from the distillation of coal tar, and their purification and conversion into intermediates by the fundamental operations of intermediates chemistry, viz. sulphonation, nitration, etc. Sections on rules of substitution and the mechanisms of reactions are included. The remainder of the volume is devoted to useful tables (*a*) summarising reactions of certain classes of intermediates, e.g. halogen derivatives, and (*b*) showing, with structural formulae, the intermediates derived from benzene, naphthalene, and anthraquinone, indicating at a glance their relationships and the routes used in their preparation.

Volume II is concerned with the chemistry of synthetic dyes, and these are dealt with according to the usual group procedure, i.e. nitro, nitroso, azo, etc. The methods of preparation, with formulae, of the different types of dyes are outlined, their principal properties indicated, and examples of important dyes, with structural formulae, are noted.

A list of books covering the field of synthetic dyes generally is given early in Volume I; references to literature dealing with particular aspects of dyes and dyeing are adequately dealt with under appropriate sections.

This publication fulfils admirably its primary purpose of providing students with lecture notes; moreover, the notes are full and comprehensive enough to constitute a modern summary of the field of synthetic dyes and intermediates, and as such deserve and should command a wider circulation among workers in this field. The space provided for the insertion of notes and references particularly enhances the usefulness and value of the work.

E. J. Cross

Lithopone

By E. A. Becker. Pp. 154. Stuttgart: Berliner Union. 1957. Price, DM 32.00.

This German book is particularly welcome, since so little has been written during recent years about zinc sulphide pigments, and much of the older literature is either rather unreliable or out of date.

The scope is considerably wider than might be inferred from the title. Thus, theories of pigmentary effect are discussed, and the relationship is traced between pigment and medium refractive index differences and pigment particle size, on the one hand, and light scattering and internal reflection, on the other. Particle sizes of various pigments to give optimum pigmentary strengths in linseed oil are listed.

The history of lithopone is traced from the two patents of the Frenchman, G. F. de Douhet, in 1847. High-strength lithopones are attributed to the Belgian, V. Leger, in 1869. J. B. Orr is mentioned as the first manufacturer in England, in the 1870s. The author implies that Orr made high-strength pigments, whereas his patent of 1874

actually covers the complete range of zinc sulphide contents from blanc fixe to zinc sulphide, and his product was mainly 28–30% lithopone.

German manufacture dates from the 30% lithopone (Schöningen Permanent White) of C. Bourjau, around 1877, and its development is traced through Dr. Rudolf Sachtleben to the present-day members of the Lithopone Kontor.

The manufacture of lithopone is described in detail with the aid of a flow sheet and good photographs and diagrams of plant. The German industry is unusual in being so largely dependent on zinciferous pyrites, from Meggen, as a raw material. Chloride roasting and extraction gives a solution containing zinc, sodium, chloride, and sulphate ions. This is purified with chlorine and zinc dust, and precipitated with barium sulphide solution to give products with high zinc sulphide contents. If sodium sulphate is first removed, by crystallisation, the zinc sulphide content may be lowered.

The effect of sodium chloride in lowering the temperature of calcination is described, and optimum calcination conditions for maximum pigmentary value are illustrated in terms of reducing powers and refractive indices. Photomicrographs show growth in size. Equipment for grinding, classification, drying, and final disintegration is discussed.

Zinc oxide contents are higher than with British products, about 0·5% for the normal (R) grade and about 0·3% for the special enamel (L) grade.

Light fastness is obtained by addition of 0·02–0·5 part of cobalt per 1000 of zinc, prior to calcination.

Uses in common products, such as paints, linoleum, rubber, oilcloth, paper, plastics, etc., are dealt with, and the final chapters give detailed methods of chemical analysis and for testing physical properties.

In conclusion one should add that the book is well written, in an easy style, and that it contains a wealth of information, much of which should prove of interest to users of all types of pigments.

J. G. Rigg

Synthetic Detergents

A Study of the Development and Marketing of a New Product

By P. A. R. Puplett. Pp. 241. London: Sidgwick & Jackson Ltd. 1957. Price, 42s. 0d.

One of the technical developments which has made so noisy an entrance on the scene that many domestic and civic patterns have been irrevocably changed has been the introduction of the synthetic detergent designed for domestic use. Inspired essentially by the economic difficulties of the first World War, and depending on the great growth of industrial aliphatic chemistry in the oil industry in the interwar years for the emancipation of detergency from fat chemistry, it took a second World War to move the emphasis from industrial to domestic applications. To one whose first job in 1924 included a scrutiny of the *Seifensieder-zeitung* to keep track of new products for the benefit of the well known soapmaker who employed him, the conservatism of soapmaking firms in refusing to take synthetic detergents really seriously for so long is a continuing source of wonder.

In the textile industry we are vitally interested in the chemistry and the properties of surface-active products, and many ably written books are available to guide us through the intricacies of constitution, synthesis, and physical properties. This book breaks fresh ground and is all the more interesting and refreshing to read in that it concentrates on the economic development of the products, although— and this is an added refreshment— the author, presumably an economist, has a useful knowledge of the chemistry of the subject. Only those who have tried know how hard it is to sort out and then set out concisely and informatively the earlier history of soaps and detergents from the bits and pieces that appear in various textbooks. Here it is done well for soaps, and also for synthetics from 1916 onwards, but the story of the sulphonated oils and the Twitchell reagents that forms a link between the two is hardly touched on.

The second portion of the book, a study of the factors which influence demand and production under present-day conditions, is prepared for in various ways. There is much information to show the great post-war rise in production both absolutely and in relation to soap. There are a most useful history of technical developments and a lively account of the standing and inter-relations of the large producing firms. There is one of the clearest statements of the requirements of a domestic detergent and the details of formulation that can have appeared anywhere, and there is a candid story of the vicissitudes through which some branded products have passed in the last ten years.

Much of the second portion is taken up with an account of the methods, effects, ethics, and economic results of advertising, especially in the relation of advertised claims to actual virtues. The author is surprisingly frank in his comments and disclosures, and much of what he has to say will be an eye-opener for us poor simpletons who only *use* the products. How can any of us claim to be rational when there is so little evidence that we can withstand the fantastic pressures of the modern advertising campaign?

H. A. Turner

Die Wirkung von Waschmitteln auf die Haut

By H. Stüpel and A. Szakall. Pp. xv + 261 with 52 tables and 68 figures in the text. Heidelberg: Dr. Alfred Hüthig Verlag. 1957. Price, DM 26.00.

The action of detergents on the skin is a topic which concerns everyone, whether it is shown in the sting of a badly compounded shaving soap on the face, the swollen and reddened hands of a housewife, or the lesions and secondary infections in the skin of a process worker. Probably because all users are not familiar with the best manner of using synthetic detergents, and also because a new set of idiosyncrasies may have been revealed, these agents have been associated with increased hazard to the skin. One of the terms of reference of the

Jephcott committee reflected the public concern about this matter. Although the report of this committee and the results of various censuses by the trade seem to show that this concern is not well founded, the value of the census as compared with objective dermatological investigations may be considered as still doubtful. Before the synthetics, soap products had their problems. The injurious action of alkali on the skin, whether it be introduced *via* a cheap cold-run toilet soap or a heavily loaded washing powder, is readily anticipated, but the bad effects, known to all soapmakers, of too much soap of low molecular weight in the product, added to promote free lathering, was less readily explained.

How much definite knowledge is there on the general topic? Most of us, looking at the problem mainly as chemists, would say very little. It is, however, chiefly a dermatological one, and the present book, which is the joint effort of a chemist and a dermatologist, claims, probably with complete justice, to be the first monograph on the subject. The bibliography contains the surprising number of 534 references, of which the greater part come from medical journals.

In the preface we are warned that "the action of detergent solutions on the skin is many-sided and complicated, and the methods of investigation very various, to which may be ascribed, among other things, the numerous contradications in the results". With all these difficulties, however, the authors have written a clear, thorough, careful, and interesting book, and although its significance may be greater to the physiologist than to others, lack of detailed medical knowledge need not deter the tinctorial chemist from finding cogent and useful information. As the field is likely to be unfamiliar to most readers of the *Journal*, the procedure of enumerating the chapter headings, not generally to be recommended for a review, will be followed.

The first section of the book (*The Human Skin*) has chapters on— morphology and physiology, submicroscopic fine-structure of human keratinous epidermis and physiology of the skin as a basis for the understanding of its protective action. The last is very detailed. The second section (*The Action of Washing Agents on the Skin*) outlines the chemical constitution of the common detergents and has long and detailed chapters on methods for assessing and investigating the reactions of the skin to detergent solutions and on the relation of particular types of washing agent to the behaviour of the skin (including such topics as the action of the synthetics, industrial dermatitis, normal and prolonged action, and toxicity). There is then a chapter which deals with specific chemical and physical actions which occur in the skin itself, including changes of pH, swelling, irritation, protein fixation, fat removal, and abrasive action. These factors are related, as far as can be done, with the constitution of the agent, and there is an incidental comparison of the soaps with the synthetics.

The final section (*Protective Agents and Preparations with Emollient and Preservative Action*) is short, reflecting the fact that only comparatively recently has much attention been devoted to the subject. Besides direct protection it also deals with treatments whereby dermatitis is avoided or mitigated by the cleansing of the skin from other kinds of substance which might be injurious. There is an account of the relation of this protective and preservative action to the constitution of specific compounds and also formulations of industrial creams and cosmetics.

With a book covering this great range of subject-matter it is difficult to give a fair impression from a few examples of the contents. One of the most interesting items is the experimental procedure for investigating changes in the different layers of the skin, and the demonstration of the action of an internal barrier against the inward diffusion of many injurious substances. The manner in which the skin reacts to restore its natural pH after disturbance makes fascinating reading. A number of photographs of the skin in normal and morbid states bring out clearly both the complexity of the subject and the difficulties of quantitative assessment. On the whole, the earlier evidence that the synthetics present no increased general dangers is confirmed.

It is reassuring to know that, although so much more investigation of the whole subject of detergent action on the skin still needs to be done, substantial progress has already been made.

H. A. Turner

Das Verhalten der direktziehenden Farbstoffe gegen tierische Fasern, Cellulose- und Kunstspinnfasern

By H. Gerstner. (No. 7 in the series: *Publications in the Field of Fibre Research and Textile Technology, Institute for Fibre Research and Technology, German Academy of Sciences, Berlin*). Pp. 300 + 54 figures in the text. Berlin: Akademie-Verlag. 1957. Price, bound DM 28.00.

The title of this book attracts attention and excites anticipation: the reading, for English readers at least, is likely to create some disappointment. As Dr. Gerstner truly says in his preface, there has been, in German, no serious attempt to bring together the results of investigations into dyeing processes since Valkó's *Kolloidchemische Grundlagen der Textilveredlung* first appeared in 1937. To this statement should be added the further one that Valkó devoted a few chapters only to dyeing in his larger treatment of textile processing, and that for some eight years or so the English reader has had "Vickerstaff" at his disposal, a book which, itself, was born out of a rapid clarification and theoretical integration of much precedent work on dyeing mechanisms. In his book, Vickerstaff acted as interpreter as well as recorder and, probably for this reason, he has so far kept a unique and distinctive place among the authors of dyeing literature. One is therefore especially eager to find out what line will be taken by later entrants into the field and whether fruitful new approaches are opening out.

The scope of the present work is different from that of most of its predecessors because it is restricted to those dyeing processes in which substantivity plays a major rôle, although, quite properly, almost every manifestation of this property that has been reported receives notice.

The ground upon which the distinction between substantive and other types of dyeing is made and the ostensible motive for the book itself resides in the statement, which many will find surprising: "Whereas the main theoretical problems of dyeing with vat dyes and with mordant dyes, the dyeing of cellulose esters and the formation of dyes in cellulose fibres by coupling could be regarded as solved some considerable time ago, there was lacking an authoritative explanation of the behaviour of directly substantive dyes towards animal and vegetable fibres". Later on, in the preface, it is made clear that the relations between substantivity in animal and in vegetable fibres are to be sought and that the way of doing this is to put before the reader all the evidence that can be collected and appears to be relevant, citing the contents of papers fairly completely for the benefit of those without immediate access to the originals.

The total result is not particularly happy except in the provision of a fairly comprehensive source book. An enormous amount of historical data is reviewed with little regard for the fact that later workers have sorted it out already, have been inspired in their own advances by some of it, and have rejected the rest as inapplicable or without significance. Of course it is possible that someone will come along and see in a neglected fact of 1904 an answer to one of his problems, but this is a fairly remote possibility if that fact has been collected by an industrious but uncritical author into a 1957 textbook.

The writing of this book stopped, and apparently stopped irrevocably, in 1954, and there are relatively few references later than 1950. In the section dealing particularly with cellulosic fibres this has led to a tragic anticlimax, since, after the tremendous grinding out of all the early evidence, the light that began to dawn in the middle forties and led, for instance, to clarifying theories such as those of C. Robinson and Derbyshire and R. H. Peters, by now familiar, has been completely missed. It is not easy to tell whether Dr. Gerstner is himself quite comfortable with the theoretical developments of modern times, for he cites them rather than explains their significance. For instance, it is difficult to tell from his treatment just what Crank's work means in the general theory of cellulose dyeing.

The investigations that have been made on the dyeing of wool by acid dyes, particularly those which establish circumstances in which electrovalencies operate, are recounted in great detail, but again the reader is not greatly assisted in finding his way through the labyrinth, often, it must be admitted, a difficult one for anybody to navigate.

Because there is less ancient history for regenerated protein fibres and for the synthetics, and because much of their behaviour is foreshadowed by that of the traditional fibres (if secondary cellulose acetate can be included with these), the treatment is more satisfactory and a great deal of information which it would be laborious to collect is made available for the reader. As an example of the difficulties necessarily attendant upon compressed accounts may be cited the description of the work of Elöd, Remington and Gladding, O'Briain and R. H. Peters, and Palmer upon the overdyeing of nylon. If one already knows about this, one must concede that the statement is accurate, but, for a beginner, how difficult to comprehend and how much clearer it would have been with a couple of graphs!

There are a few mis-spellings of proper names, but references are generally complete and accurate. In a book so compendious, the omission of both subject and author indexes must be counted a grave defect. It is distasteful to receive so coolly a book into which much hard and honest work has been put, but perhaps, in a later edition, the breath of life will be blown into it, and it will become a real source of inspiration to its readers.

H. A. Turner

Biological Staining Methods

By G. T. Gurr. 6th edition 1957. Pp. vi + 102 + 3 colour plates. London: George T. Gurr Ltd. Price, 5s. 0d.

In the introduction to this booklet the author lists the apparatus necessary to a microscopist who wishes to employ biological staining techniques, with some of the precautions necessary for the storage and application of stains. A section is devoted to embedding materials and mounting media, and this, together with the introduction, should prove useful to new workers in the field who are unfamiliar with standard methods.

The remainder of the book is a catalogue of some 130 stains, and sections are included on bacteriological and haematological stains and the general uses of stains. Under each staining reaction the solutions required for the test in question are given together with an annotated list of staining procedures and results of each test.

A small section of the book is devoted to the newer technique of fluorescence microscopy, which is becoming increasingly important in biological and related fields of research.

A useful formulary is appended which gives details of the constituents of the various stains listed in the booklet, and conditions for their preparation. Tables of stain solubiliites and refractive indices of the various mounting media used in the field are also included.

The booklet is well laid out, and its price does not do full justice to its contents. It is a valuable contribution, and should be a useful reference work for students, experienced technicians in this field, and all concerned with microscopical staining.

D. H. Rawson

Proceedings of the

Work Study Conference

for Senior Work Study Practitioners and Works Managers

Organised by the Association of British Chemical Manufacturers at Harrogate, 23rd–25th May 1957. Pp. v + 206. London: A.B.C.M. Price, 13s. 6d.

As the title implies, this report contains a wealth of information, not only from the contents of the various lectures delivered by people experienced in both management and work study, but also from the constructive criticism offered during the discussion periods, all of which are fully reported. These proceedings emphasise the many advantages gained by the use of work study but also point out some of the problems arising from such work in practice, and the limitations of the various techniques employed. The contents of this report can be considered under three main heads— the human implications to both management and men of the application of work study, the application of incentive schemes both to process workers and to engineering maintenance workers, and work study in new design. The greater number of papers deal with incentive schemes, and are mainly technical in character.

Experienced work study officers and managers who have had control of labour on applied work should derive benefit from this report, which offers better perspective and understanding in work study matters. Such papers as those given on personnel problems, and the final lecture on *Work Study in the Smaller Factory*, will be of wider interest to the person wishing to know something of the effects of work study on the chemical industry.

Human relations in the application of work study was the theme of the first two papers— one by a works director on staff problems, and the other by a trades union official on the trade union attitude to work study. Both speakers emphasise the need for adequate training, communication, and co-operation at all levels from the managing director to the hourly paid worker. An interesting discussion on the advisability of workers checking work study values suggested that this was of limited value, while the general trend of thought on method studies favoured a team approach of chemist, engineer, and work study officer to all method studies of chemical processes and engineering plant.

The papers given on *Work Study in Design* and the *Use of Models in the Design of Chemical Plant* describe some results of the work study approach to problems of this nature, and make a comparison of this method with the same work undertaken by traditional methods. The need to consider maintenance and manning of a plant in some detail at the design stage was also stressed.

The remaining lectures are mainly concerned with the technical application of incentive schemes, and this (as would be expected) was the most controversial of the subjects undertaken. They cover situations from single-product plants, multiproduct chemical factories, to engineering maintenance schemes, and the merits and demerits of proportional, geared, and multifactor schemes, as applied to chemical work, are discussed. Some interesting comments are made on the problem of the assessment of mental work carried out by an operator.

The publication of this interchange of views on work study by such a wide section of the chemical industry provides a useful addition to the technical information already available on this subject.

W. Hesketh

Notes on Work Study No. 3

Case Histories from Smaller Firms and Individual Plants in the Chemical Industry

London: Association of British Chemical Manufacturers. Pp. v + 41. [1958.] Price, 4s. 0d.

This is the third in a series of Work Study Booklets, published by the Association of British Chemical Manufacturers. The text is subdivided into three sections, two of which give the results achieved by the application of method study techniques to both chemical processes and the ancillary work connected with such processes. Examples given include the handling and packing of materials and the maintenance of plant units, in addition to studies on chemical processes. The third section describes case histories of incentive schemes and the work carried out on small-plant processes, and embraces the more specialised field of work measurement. The case examples have been chosen with care, not only to show that labour costs have been reduced, but to illustrate that increased productivity has been achieved by the use of different equipment, changes in process, better plant utilisation, and improved quality standards. The method studies were carried out by work study officers in some cases, but in others chemists and engineers were also employed either singly or as members of a team. Finally, these examples show that appreciable improvements were made, varying from an increase in production of 150% with reduced labour costs in one example, to a saving of £5,000 per annum in another. The use of work study in engineering maintenance work is also emphasised.

This booklet fulfils its function in showing that small manufacturing units can derive great benefit from the application of these techniques. It lays bare the fallacy of the supposition that only large firms can afford to carry out such studies, while at the same time making the necessary reservations on the use of these techniques where applicable. Like all condensed literature it may, however, appear too simple, and some people may well be inclined to attempt to apply these techniques without first getting to understand the fundamentals of the subject. Results only are given, and no attempt has been made to point out the practical difficulties in their application, or the importance of such factors as human relations in the achievement of these results.

Technical management may criticise it, in that some of the results reported under the heading of work study could better be described, for example, as the work of the experimental chemist. More emphasis is perhaps needed in pointing out that

method study charting and the critical analysis of these charts is an aid whereby a more thorough appreciation of the possible alternative ways of doing work can be obtained. It is therefore an additional aid in the quest of better management, and does not replace these other functions.

The reference to incentive schemes shows that further increased productivity can be achieved by the application of such schemes, but the warning that they must be based on sound work study principles is very necessary. Whereas the production team can be readily trained to use method techniques, incentives and work measurement techniques require more specialisation and experience, and as a result this section may be read with less interest than the rest of this admirable booklet. A bibliography to encourage further reading is also given.

W. HESKETH

Ion Exchangers in Organic and Biochemistry

Edited by C. Calmon and T. R. E. Kressman. New York: Interscience Publishers Inc. 1957. Pp. xii + 761. Price, $15.00.

With the increasing use of ion-exchange resins in all fields of chemistry, this book comes as a valuable contribution in correlating the large amount of information available in the various journals.

The book is divided into three parts. The first deals with the production of the different types of resins and theoretical aspects of their reactions. The second part discusses the commercially available products and the laboratory techniques for their application. The third part, which considers specific applications of the resins, both inside and outside the laboratory, comprises nearly two-thirds of the book. This part should be of interest to any chemist or biochemist, because the uses selected are so diverse that, whatever the reader's field of work, he is almost bound to find something of value. In general interest the book is fascinating. Most chemists are well aware of the value of resins in the separation and analysis of materials, but the uses given range from the isolation of virus particles and the treatment of metabolic diseases to their value as catalysts in organic reactions. The importance of ion exchanges *in vivo* is well and interestingly described. The book ends with a concise chapter on the use of resins in water treatment. This seems somewhat mundane after the previous chapters, but it is a timely reminder that this is still one of the most valued uses in industry.

All the chapters are written by experts, who in the main have given a readable account of their speciality. All give a good bibliography of their particular field.

Dyers will be interested to know that wool is a "multiheterofunctional natural resin". More important, however, is the treatment of resins and cellulose to introduce acidic and basic groups, which would be interesting in the dyeing field.

The book, like most American publications, is well printed and, again like most, is expensive. It is most interesting and enjoyable to read, and has a considerably closer association with dyeing than might be expected.

R. S. ASQUITH

New Books received

Guida dei Principali Prodotti Chimici. CESARE FERRI. Volume II. Bologna, Italy: Nicola Zanichelli Editore. 1958. Pp. xv + 650.

A Vocabulary of Technical Terms used in the Textile and Related Industries. English–French, French–English, English–German, German–English. Manchester: Imperial Chemical Industries Ltd. 1958. Pp. 103. No price.

The Textile Recorder Annual 1958–1959. Book of the Year. Manchester and London: Harlequin Press (1955) Ltd. [1958.] Pp. 172. 30s. 0d.

Conference Terminology. A Manual for Conference-members and Interpreters in English, Russian, French, Italian, Spanish, German. J. HERBERT (editor). Amsterdam: Elsevier Publishing Co. 1957. Pp. xv + 147. 12s. 6d.

De Biochromaatbeitsing en de Kleurbaarheid van Gebeitst Lipoid Onderzocht met Behulp van Reflectometrie. Met een discussie over de Photometrie van Kleurstofvlekken. (Proefschrift ter Verkrijging van de Graad van Doctor in de Geneeskunde aan de Rijksuniversiteit te Leiden . . . 12 Juni 1957). F. J. M. HESLINGA. Pp. 173.

Drycleaning. Technology and Theory. A Report of the National Institute of Drycleaning. A. R. MARTIN and G. P. FULTON. New York: Textile Book Publishers Inc. A Division of Interscience Publishers Inc. 1958. Pp. viii + 269. $6.00.

Le Mécanisme de la Vision des Couleurs. Physiologie–Pathologie. J. SÉGAL. Paris: G. Doin & Cie. 1953. Pp. 351 + 1 colour plate. 3,200 francs.

Mechanismus des Farbensehens. Physiologie–Pathologie. J. SÉGAL. Jena: VEB Gustav Fischer Verlag. 1957. Pp. xv + 283 + 1 colour plate. DM 32.00.

Supplementary List of Publications of the National Bureau of Standards. 1 July 1947–30 June 1957. Washington: United States Government Printing Office. 1958. Pp. iv + 373. $1.50.

Report to the Worshipful Company of Clothworkers of the City of London of the Advisory Committee on the Departments of Textile Industries and Colour Chemistry and Dyeing in the University of Leeds. Session 1954–1955. Pp. 46. [1955.] Session 1955–1956. Pp. 46. [1956.] Session 1956–1957. Pp. 47. [1957.]

Précis des Matières Colorantes Synthétiques. Tome I—Matières premières et Produits intermédiaires. HENRI WAHL. Paris: Presses Universitaires de France. 1958. Pp. iv + 330. 2,400 francs.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Ciba Ltd.

Adhesive FD Ciba— A colourless, permanent adhesive, based on synthetic resin, recommended for gumming down in screen printing.

Melafix II— If this product is used as a buffer in the chlorination of wool it is possible to add all the hypochlorite to the bath before entering the goods. The degree of shrink-resistance achieved and the uniformity of chlorination are as good as when Melafix CH is used, and the total time, including anti-chloring and softening, does not exceed 90 min.

Cibacet Dark Blue RB— This disperse dye gives dark blues on polyester and polyamide fibres and rather more reddish blues on secondary cellulose acetate and cellulose triacetate. On polyester fibres it has good build-up properties and gives dyeings of good fastness to light. On cellulose triacetate it shows particularly good fastness to pleating, and it has the same fastness to light on both bright and delustred secondary acetate. It is suitable for direct printing on all the fibres mentioned above. Fastness figures on Terylene include— Light (for a dyeing produced using o-phenylphenol) 5–6, washing b 5, pleating (Rabofsky 170–210°c., 60 pleats per min.) 3–4 (staining of white Terylene).

Cibacron Dyes in Dyeing— This card describes the dyeing characteristics and methods of application of eight Cibacron dyes and gives full details of the fastness of dyeings on cotton and spun viscose rayon staple both with and without a crease-resist finish. A novel feature is the inclusion of infrared inflectance figures for 40 g./litre dyeings on cotton poplin. Each dye is represented by dyeings in five depths on mercerised cotton cloth produced by the pad–steam process, and the behaviour of each with nine different fibres including Orlon 42, Terylene, and Courpleta is shown by pad–dry heat dyeings on a composite material. White discharges of dyeings of seven of the dyes are also included. Forty dyeings of mixtures of Cibacrons are presented. The materials used are continuous-filament and spun staple viscose rayon, linen, cotton, and jute, and the methods of application include pad–jig development, pad–steam, pad–dry heat, and conventional dyeing on the winch.

Direct Dyes on Cotton and Viscose Rayon— This card contains dyeings in three depths on cotton and spun viscose rayon staple cloth of 94 direct dyes. In addition there are dyeings on continuous-filament viscose rayon crêpe in three depths of eight Rigan and Rigan Fast dyes designed to give solid dyeings on viscose rayon having unequal dye affinity. The now customary exhaustive compilation of data on dyeing behaviour and the fastness characteristics of dyeings are included together with time–temperature curves for each dye.

Vibatex K; Vibatex K conc.— Vinyl resin emulsions for bonding, coating, and special finishes.

The Geigy Co. Ltd.

Graphical Illustrations of the Dyeing Properties of Wool Dyestuffs— This volume includes graphs representing the dyeing and migration characteristics of 242 wool dyes classified as follows: level-dyeing acid dyes, light fastness of 1/1 standard depth dyeing < 5, 37; similar, but light fastness > 5, 36; weakly acid-dyeing dyes 36; Polar dyes 30; Irgalan dyes 23; Irganol dyes 4; Enolan (strongly acid-dyeing metal-complex) dyes 8; chrome dyes 68. For each dye two curves have been drawn. In the first, dye uptake is plotted against time of dyeing, the temperature being raised at a constant rate to 100°c. in 75 min. and dyeing continued at 100°c. for a further 60 min. in all cases except with chrome dyes, where dyeing was continued at 100°c. for a further 30 min. after adding dichromate. In the second the amount of dye remaining on material originally dyed to 1/1 standard depth and the amount transferred to white wool are plotted against time of immersion in a boiling bath containing the same assistants as used in dyeing. In each case separate curves are drawn to represent each set of dyebath conditions of interest when applying a particular dye and, in particular, to emphasise the effect of pH on absorption and desorption. The text includes a description of the purpose, experimental basis, method of interpretation, and uses of the graphs. This volume provides the wool dyer with detailed and precise information on the behaviour of his dyes in the same form as that made available to the dyer of cellulosic materials in *The Dyeing Properties of Direct Dyes* published in 1944 (2nd edition 1948).

Imperial Chemical Industries Ltd.

Wool Slubbing: Chrome Dyestuffs Fast to Light and Milling. Volumes 1 and 2— The two cards contain dyeings on wool slubbing in five depths (three for navies and two for blacks) of 39 chrome dyes. The dyeings, which have been produced by the recommended process for the particular dye, are mounted on loose leaves in the now standard fashion. The suitability or otherwise of each dye for application at temperatures > 100°c. is included in the data provided. A major change is the substitution of light fastness figures for dyeings in "standard depth for pale shades" (equal to $\frac{1}{50}$–$\frac{1}{100}$ standard depth) instead of those previously used (equal to $\frac{1}{4}$–$\frac{1}{16}$ standard depth). Solubility figures are quoted (at 90°c.) for the first time.

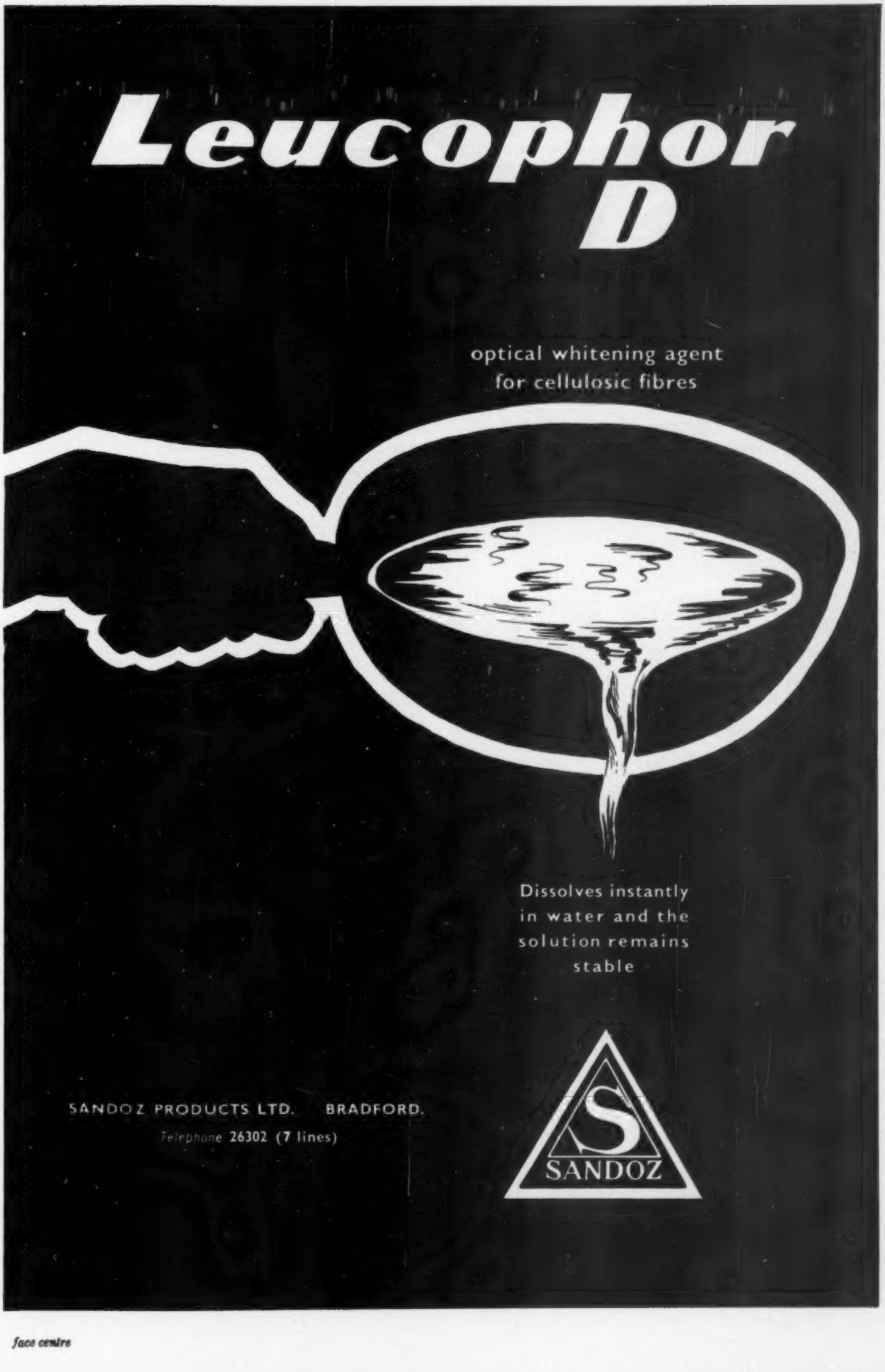
Procion Dyestuffs in Textile Dyeing: Natural Silk and Nylon— This loose-leaf type of card contains dyeings on nylon staple fibre yarn and spun silk yarn of nine reactive dyes from the Procion range. They may be applied to silk either from an alkaline (soda ash) dyebath at 20–50°c. in presence of Glauber's salt or from a formic acid dyebath at 85°c. The former is generally preferred. The less reactive H type do not build up as well and are not suitable for dyeing from acid baths. A feature of the acid-dyeing method is that, when it is used with silk–cellulosic fibre mixtures, the latter is reserved. The more reactive, cold-dyeing Procions may be applied by the continuous methods recommended for cellulosic fibres. Nylon staple (as loose fibre, slubbing, yarn, and cloth) may be dyed with Procion and Procion H dyes from a formic acid dyebath at the boil. They are not recommended for dyeing continuous-filament nylon, since they do not cover yarn irregularities. Their levelling power is low; uniform initial uptake of dye is essential, and careful control of dyeing temperature must be exercised. Mixtures often show some incompatibility.

Sandoz Products Ltd.

Cuprofix Brilliant Blue C-2BL— This direct dye gives bright blues on cellulosic fibres when aftertreated with either copper sulphate–acetic acid or Cuprofix SL. Dyeings have good fastness to light and wet treatments, and are suitable for urea–formaldehyde crease-resist finishing. Secondary cellulose acetate is reserved practically white. Fastness figures for a dyeing aftertreated with copper sulphate include— Light 6, washing (60°c.) 4–5, perspiration 4–5.

Lanasyn Brilliant Yellow 5GL— This neutral-dyeing acid dye gives bright yellows on protein and polyamide fibres and is intended for use alone and as a brightening colour in mixtures with Lanasyn and other fast-to-milling dyes. It is applicable to loose fibre, yarn, and cloth to give dyeings of very good fastness including fastness to decatising, stoving, and acid and alkaline milling. It is stable to chrome in the dyebath and is of interest for medium-depth dyeings on wool–nylon mixtures. Fastness figures on wool include— Light (daylight S.N.V.) 6, washing (60°c.) 5, heavy milling 4–5.

Lanasyn Colours on Wool Piece— This card contains dyeings in three depths on wool cloth of twenty neutral-dyeing metal-complex dyes of the Lanasyn range and five Lanasyn Brilliant dyes (including a black) which are compatible with them and recommended for shading. The scope of these two groups of dyes is further illustrated by a range of 150 mixture dyeings based on 30 different two- and three-colour combinations. The text includes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optical whitening agent for cellulosic fibres
Dissolves instantly in water and the solution remains stable
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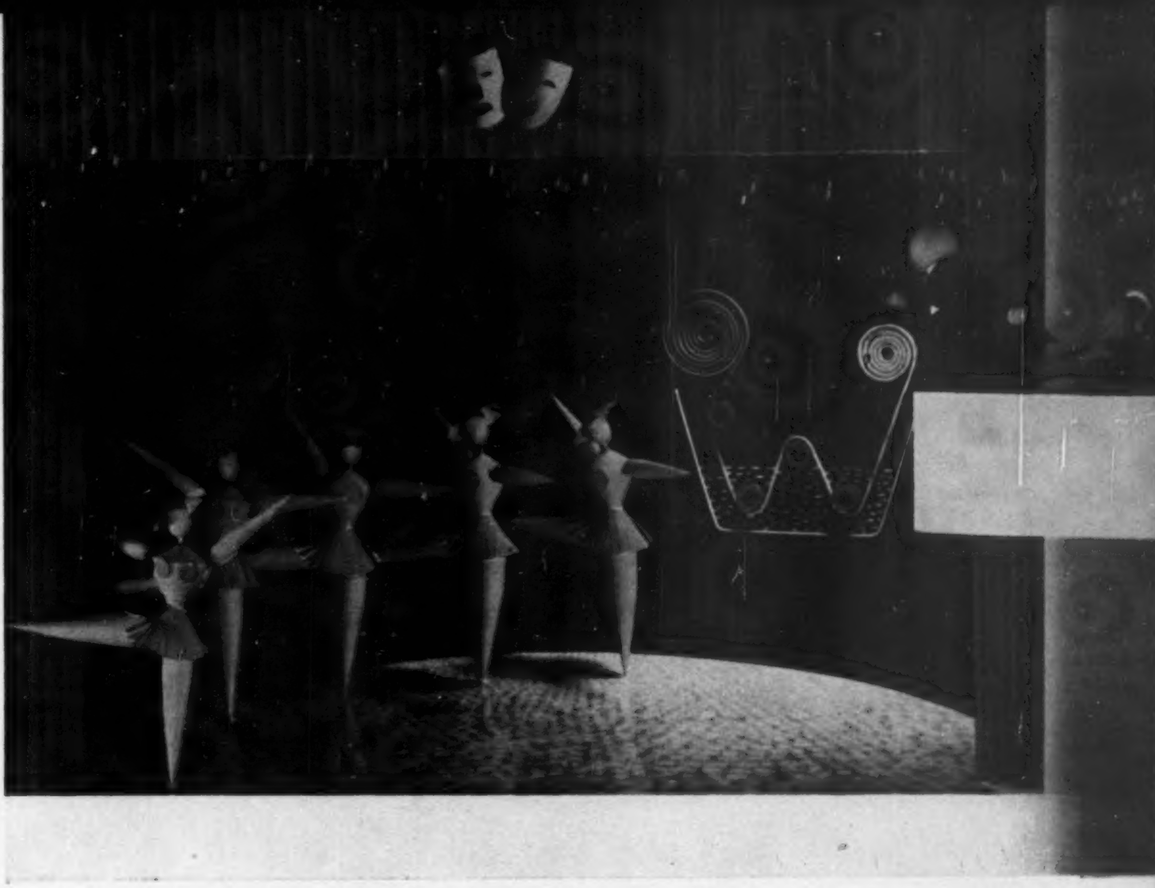
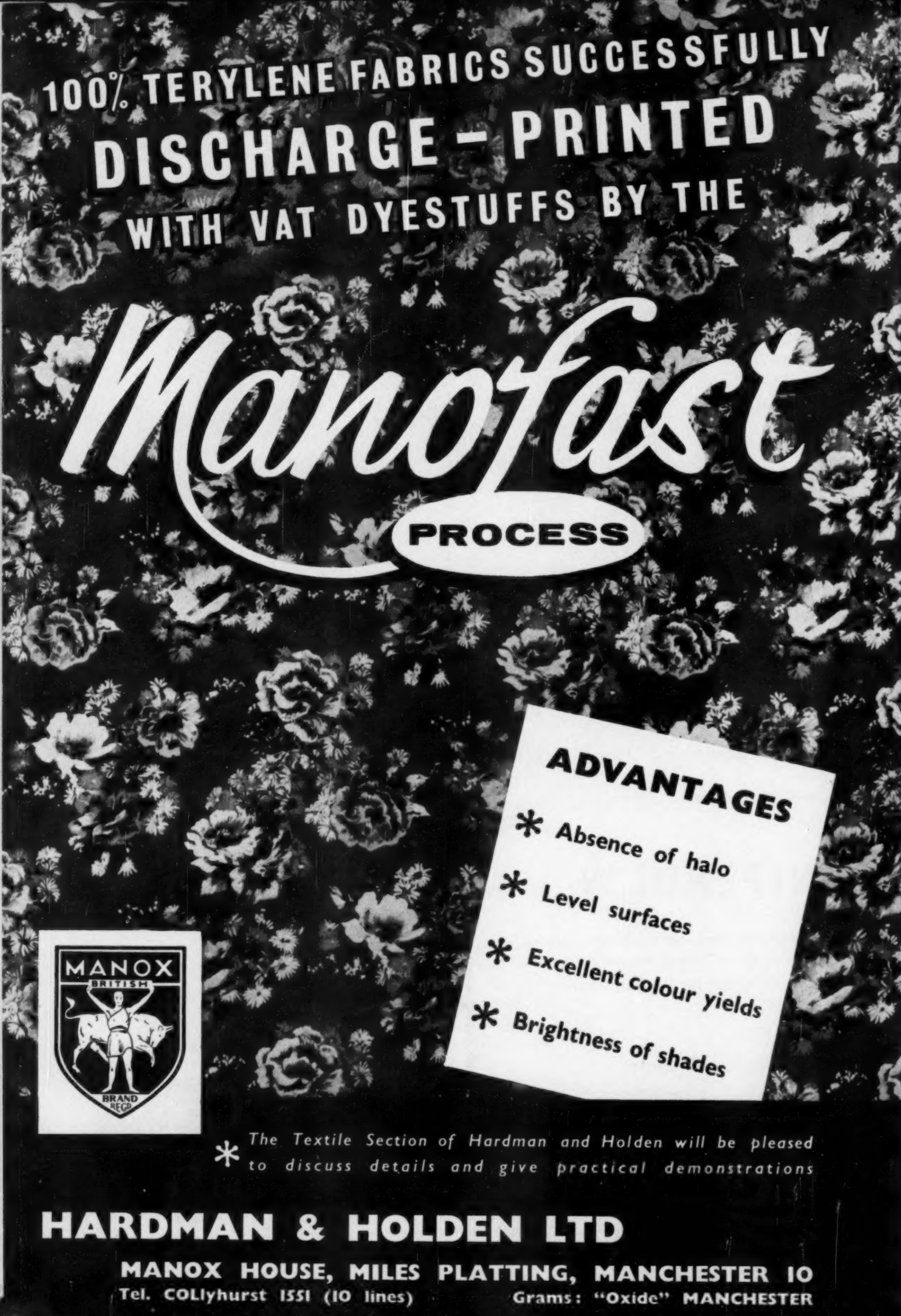
SANDOZ

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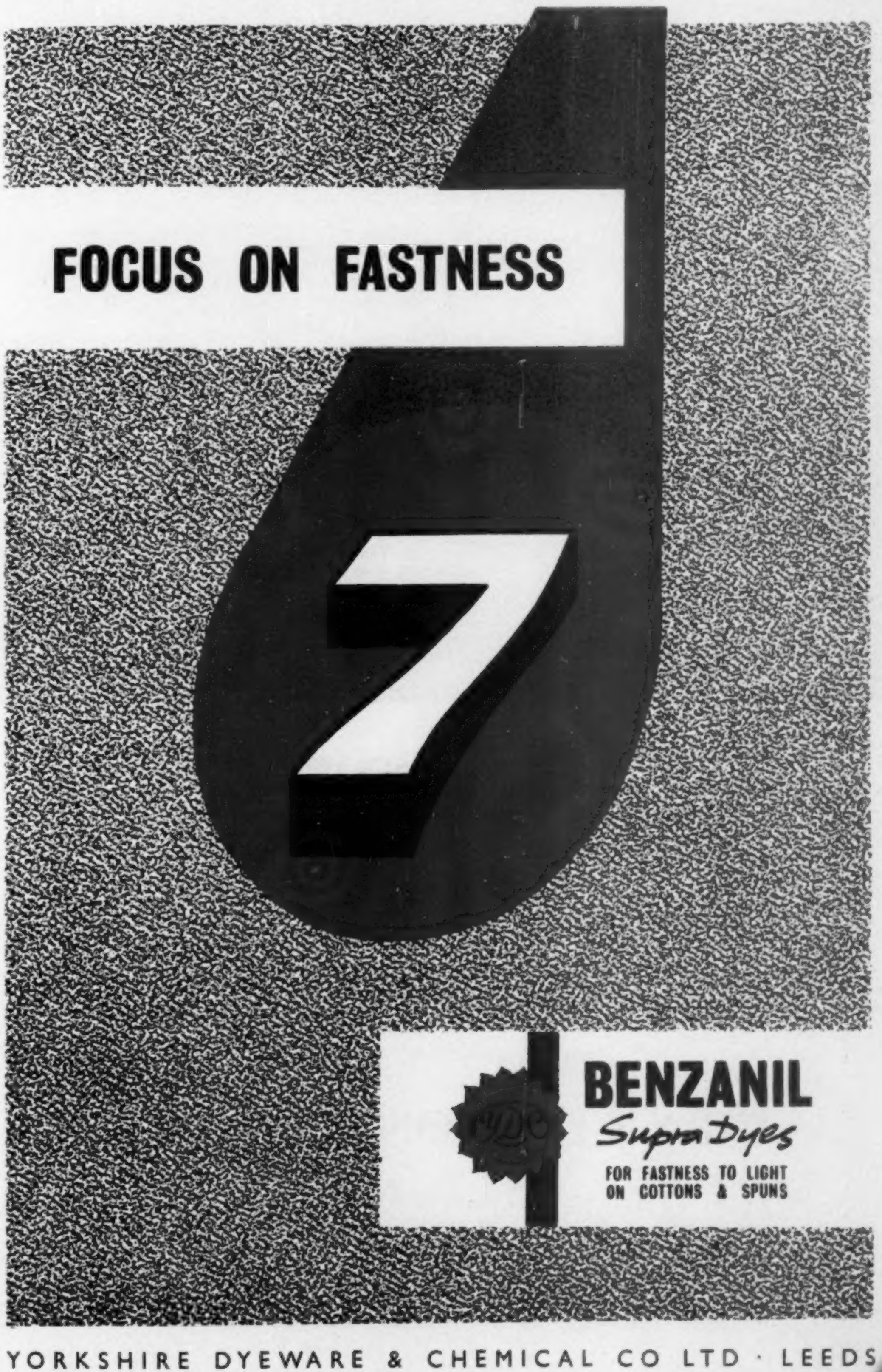
FOCUS ON FASTNESS
7
BENZANIL
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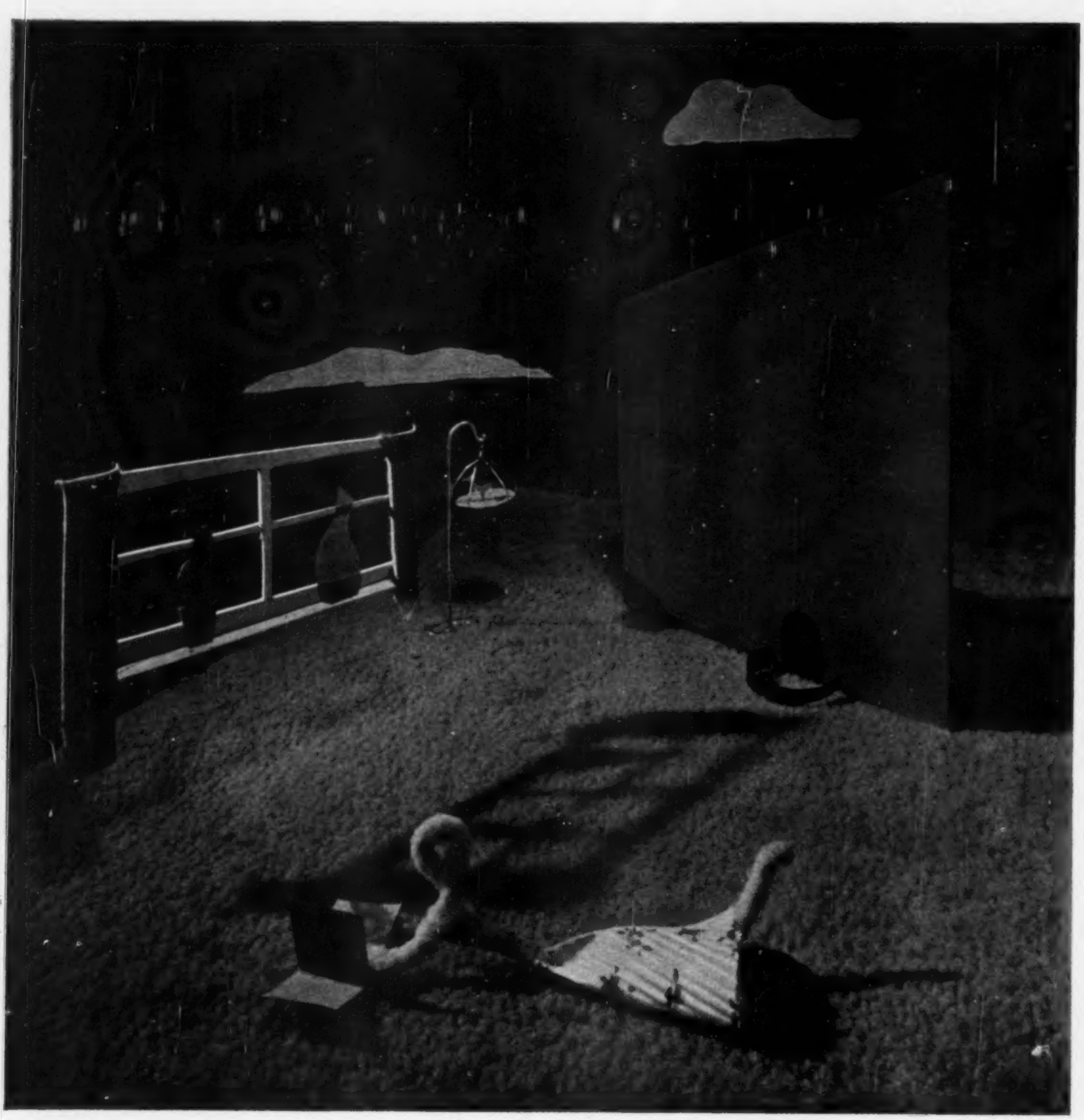

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Copper-free a range of dyestuffs to give a wide and varied selection of light-fast shades on viscose rayon

Compatible the ideal combination
Solophenyl Yellow 2RL Solophenyl Scarlet 2GL Solophenyl Blue FGL
for fawns and greys

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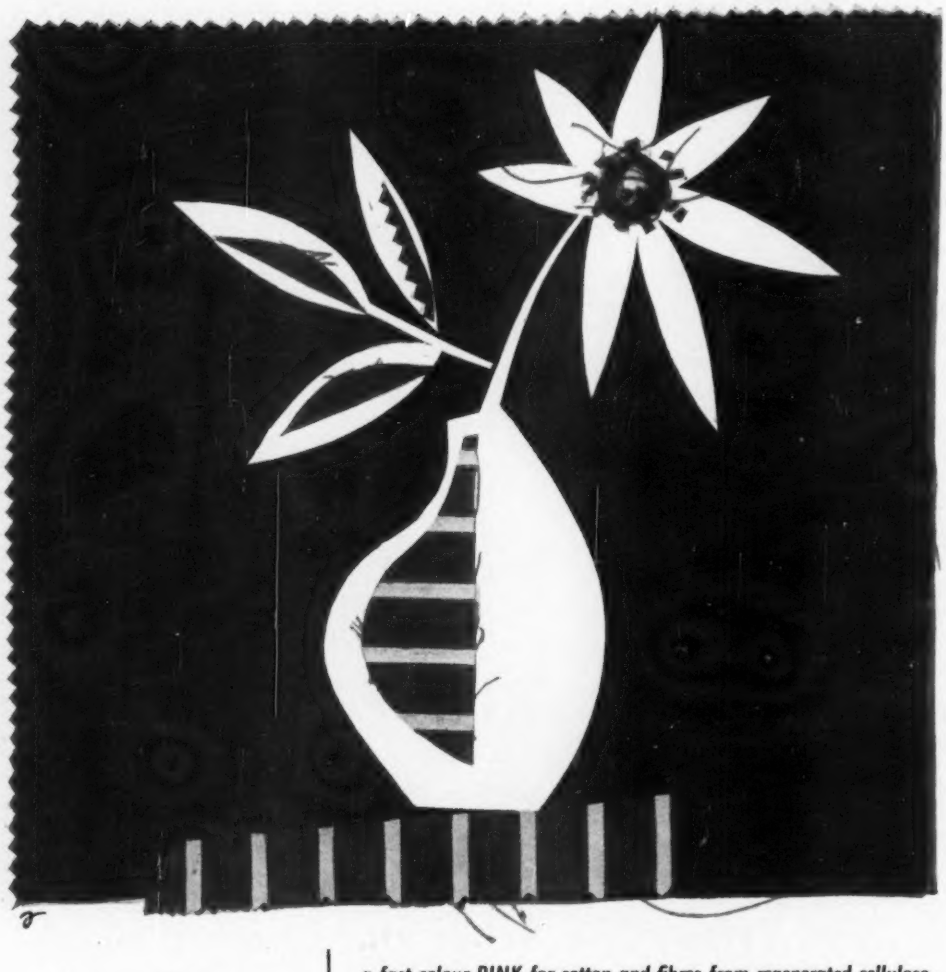


details of the acid—Lyogen SMK process for applying the Lanasyn dyes.

The Yorkshire Dyeware & Chemical Co. Ltd.

SERISOL DYES ON SECONDARY ACETATE— This card contains dyeings in two depths on bright continuous-filament locknit of 69 disperse dyes together with a single dyeing of Hemasol AR Paste. Data appended alongside each set of dyeings include suitability for use on triacetate, polyamide, polyacrylic, and polyester fibres. The additional text contains full information on the preparation and dyeing of secondary cellulose acetate, selection of dyes for special requirements, and the application of disperse dyes to other fibres and also to plastics and sheepskin fleeces. Altogether it is a very imposing volume, the completeness of the contents being matched by the excellence of the presentation.

SERISOLECTOR— This is subtitled an "instant selector of disperse dyes". The range of disperse dyes has been listed according to colour and substrate in such a way that the most suitable selection of single dyes or mixtures suitable for producing oranges and yellows, reds, violets, blues and navies, greens, browns, and greys and blacks, on any of the following— secondary cellulose acetate, cellulose triacetate, nylon, Terylene, Acrilan, and Orlon 42 and Courtelle— may be turned up directly. In most cases figures for fastness to light, washing, sublimation, and gas fumes (where significant) are given together with any important information on building-up properties.

DISPERSE DYES ON ACRILAN— This card contains dyeings in two depths (except for black) on Acrilan cloth of 12 disperse dyes, including 8 from the Serisol range, 2 Serinyl, and 2 Serilan dyes. Fastness data for daylight, washing (5 g. soap per litre at 60°C. for 45 min.), and sublimation (steaming at 10 lb./sq.in. for 15 min.) are appended together with general comments on the properties and uses of each dye.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Winch for Dye Vessel
Collins & Aikman Corpn. *USP* 2,793,034

A winch made up of bars having smooth surfaces but which can nevertheless successfully deal with heavy fabrics without slippage or damage to the fabric. C.O.C.

Winch for Dyeing Machine
Carolina Rubber Hose Co. *USP* 2,796,169

The winch is formed of elongated cylindrical members each of which has a covering with a flexible irregular surface, e.g. fluted rubber, to engage the fabric without damaging it. C.O.C.

Pressure Dyeing Machine for Fabrics
Burlington Engineering Co. *USP* 2,792,702

A machine in which cloth batched on perforated rollers is dyed at up to 300°F. under pressures of up to 50 lb. per sq. in. The machine is easily loaded and unloaded and all operations including reversal of flow of the dye liquor through the beam and fabric wound on it are automatically controlled. C.O.C.

Web Tensioning Mechanisms
Huck Co. *BP* 794,998

A simple device for maintaining to a fine degree of accuracy constant tension in a moving web. C.O.C.

Untwisting Materials in Twisted Rope Form
Bradford Dyers' Assocn. *BP* 794,429

Apparatus for untwisting roped material coming from e.g. a hydroextractor. C.O.C.

Multicolour Machine for Printing Fabrics
R. B. Low *USP* 2,789,498

Each printing roll has a doctor blade which is adjustable to vary the amount of printing paste supplied to the roll. The printing rolls are independently driven to continue rotating after the material to be printed has passed through them so that the printing paste will not dry on the rolls. Means are provided to tighten and align the feed blanket. Cloth and thermoplastic sheeting can be continuously and rapidly printed with several colours on this machine without the prints becoming smeared or blurred. C.O.C.

Multicolour Attachment to the Print Roller in Roller Printing
R. K. Sinejda *BP* 794,404

A versatile multicolour feeding apparatus easily installed in conventional printing machines. It is simple and economical to operate. C.O.C.

Application of Resin Finishes to Cotton Garments using Dry-cleaning Plant Equipment (X p. 613)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Organo-chemical Contribution to the Inorganic Chemistry of Nitrous Acid
C. K. Ingold

J. Roy. Inst. Chem., **82**, 267–268 (April 1958)

This lecture deals particularly with electrophilic substitution reactions, and it is shown that, in the reaction of an electrophilic reagent (such as nitrous acid) with a substrate which results in the transfer to the substrate of a group X to be bound by electrons supplied by the substrate, the simplest of the possible carriers of X (e.g. NO) for the transfer would be X^+ (e.g. NO^+); others are of the type X–Y (e.g. NOBr), which might be an ion or a molecule, but in which the ease of separating group Y as Y^- by the reaction $X{-}Y \rightarrow X^+ + Y^-$ would be an important factor in the efficacy of the carrier, whereas another factor would be the limiting concn. of X–Y, which might be present as such in the reaction medium. Of the possible carriers of NO in nitrosation, the following, arranged in order of increasing basic strength, were considered: NO^+, $NO{\cdot}OH_2^+$, NO·Hal, $NO{\cdot}NO_3$, $NO{\cdot}NO_2$, $NO{\cdot}\overline{Ac}$, and NO·OH.

In the catalytic nitration of phenols two types of reaction were reported, the first being kinetically of zero order and due to the well established course of nitration by the nitronium ion, while the second, arising from the catalysis and of the second order, was exemplified by reactions in acetic acid medium with nitric acid in excess of nitrous acid, viz.—

$$ArH + \text{stoichiometric } HNO_2 \xrightarrow{\text{slow}} ArNO + H_2O$$

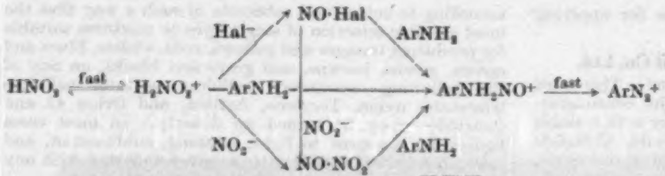
and

$$ArNO + HNO_3 \xrightarrow{\text{fast}} ArNO_2 + \text{stoichiometric } HNO_2.$$

Under these conditions nitrous acid is present mainly as the slightly dissociated N_2O_4 mol.—

$$2NO_2 \rightleftharpoons N_2O_4 \rightleftharpoons NO^+ + NO_3^-$$

so that both NO^+ and N_2O_4 are possible carriers. It was found that, whereas the NO^+ ion concn. is considerably less than that of the N_2O_4 mol., the nitrosonium ion is specifically the more powerful nitrosating agent. In the case of the diazotisation of aromatic amines in weakly acidic soln. containing small amounts of $HClO_4$ as the only strong acid, the list of possible carriers reduces to NO^+, $NO{\cdot}OH_2^+$, $NO{\cdot}NO_2$, and NO·OH. Under suitable conditions, including addition of halide ion, when NO·Hal becomes a possible carrier, the reaction was found to proceed by any of the following routes—



H.H.H.

Specific Heats of Aqueous Solutions of some Soaps
V. I. Solnŷshkin

Colloid J. U.S.S.R., **19**, 629–632 (Sept.–Oct. 1957)

The specific heats of aq. soln. of sodium oleate and ricinoleate have been determined. The presence of a flattened-out section (Davis plateau) in the concn. range 0·25–0·65 M. for sodium oleate and 2·25–2·85 M. for sodium ricinoleate soln. is attributed to the formation of laminated soaps. A mechanism for the aggregation of non-ionic soap molecules is suggested on the basis of autoflocculation to spherical micelles with subsequent transition to the plate form and the formation of a colloidal solution, from which hydrates of the soap crystallise as the concn. is increased. G.J.K.

Colloidal Properties of the Esters of Sodium Sulphosuccinate. I— Esters of the Lower Aliphatic Alcohols
R. V. Kucher, A. A. Yavorovskiĭ, and M. A. Kovbuz

Colloid J. U.S.S.R., **19**, 454–458 (July–Aug. 1957)

The dimethyl (DME), diethyl (DEE), dibutyl (DBE), and di*iso*amyl (DIAE) esters of sodium sulphosuccinate have been synthesised by esterification of the corresponding alcohols with maleic acid and addition of sodium bisulphite to the esters. The surface activity of these compounds in aqueous solution has been studied, and it is shown that DME and DEE are devoid of this property; DIAE exhibits considerable surface activity (σ = 32·5 ergs/cm.^{2} for a 2% soln.). Measurements obtained at pH 9·9 (*a*) of the critical micelle concn. in very dilute solutions of colloidal electrolytes (using the light-scattering technique) and (*b*) of the micellar wt. in solutions up to 2·5% (by extrapolating the curve $H(c/\Delta\tau) = f(c)$ to zero concn.) are used to calculate the energy of association in micelle formation (P. Debye, *J. Phys. Colloid. Chem.*, **53**, 1 (1949)). The authors show that the tendency for micelle formation is negligible for DME, DEE, and DBE, but that DIAE has a considerable micellar wt. (71,400) and a low critical micelle concn. ($3{\cdot}05 \times 10^{-5}$ M.). The conjugate solubility of Sudan III in aqueous solutions of the lower esters of sulphosuccinic acid (measured photometrically) begins to grow noticeably with DBE, and is particularly large in the case of DIAE. G.J.K.

Textile-processing Chemicals
L. D. Berger

Amer. Dyestuff Rep., **47**, P 179–P 182 and P 190 (24 March 1958)

An analysis of the categories of textile-processing chemicals and an assessment of their economic importance. Synthetic warp sizes, solvent-dyeing processes, and anti-static agents are recent developments which are discussed. P.T.S.

PATENTS

Monosulphoxides having Detergent Properties
Union Oil Co. of California *USP* 2,787,595

Compounds of formula R^1·SO·R^2 (R^1 = CH_3 or C_2H_5; R_2 = Alk of > 5 C), e.g., methyldodecylsulphoxide, have good detergent properties. They are unaffected by alkaline earth and heavy metal ions. C.O.C.

Solutions of Dextran in Aqueous Boron Trifluoride as Textile Finishes and for Producing Fibres
Commonwealth Engineering Co. *USP* 2,787,518

Dextran is readily soluble in 50–70% aqueous boron trifluoride at room temperature. The solution obtained can be used as a textile finish, e.g. cotton cloth impregnated in a bath containing 5% of native (unhydrolysed) dextran in 60% aqueous boron trifluoride, rinsed and dried, has a crisp organdie finish. The solutions can also be extruded into water to form filaments. C.O.C.

Composition Providing Available Chlorine
Theobald Industries *USP* 2,795,556

1:3-Dichloro-5:5-dimethylhydantoin or other chlorinated alkylhydantoins used in aqueous solution with a hydrotrope, e.g. Na toluene sulphonate, and an antacid buffer salt, is an excellent bleaching agent for use in laundering. C.O.C.

***NN′*-Oxydimethylene-bis-acylamides as Water-repellent Finishes**
DuP *USP* 2,793,142

Compounds of formula—

R^1·CO·NH·CH_2·O·CH_2·NH·CO·R^2

(R^1 and R^2 = same or different, straight chain aliphatic hydrocarbons of 9–29 C) are prepared by heating *N*-hydroxymethylamides at 40–75°C. in an inert vehicle containing < 3% of water on the wt. of the amide. They are used, e.g. in aqueous dispersion, for impregnating textiles in presence of an acid catalyst and then heating to 110–150°C. to obtain a water-repellent finish resistant to washing and dry-cleaning. The dispersion used preferably also contains a protective colloid and a heat-labile, transitory dispersing agent. C.O.C.

Titania Monohydrate as Soil-resistant Finishing Agent
American Cyanamid Co. *USP* 2,788,295

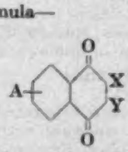
Titania monohydrate of particle size 0·02–0·075 μ. is used in 0·5–1·5% (calc. as TiO_2) aqueous dispersion.

USP 2,790,737

The titania monohydrate is employed in conjunction with a polymer of a compound containing a CH_2:C< group, e.g. stearamido propyldimethylhydroxy-ethyl ammonium chloride. The polymer is produced by emulsion polymerisation, preferably in presence of the titania monohydrate, and is washed free of emulsifying agent before application. C.O.C.

Dithiocyano-substituted 1:4-Naphthaquinones — Fungicides
American Cyanamid Co. *USP* 2,796,377
USP 2,796,425

Compounds of formula—



(A = H or nitro; X and Y each = thiocyano or thiocyanomethyl), e.g. 2:3-bis(thiocyanomethyl)-1:4-naphthaquinone and 2:3-bis(thiocyano)-5-nitro-1:4-naphthaquinone, are good fungicides for use on textiles, fur, leather, etc. C.O.C.

Readily Soluble Compositions containing 4-Methyl-7-aminocoumarin Fluorescent Brightening Agents (IV p. 603)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Light Absorption of Dyes in Solution
Y. Ooshika

Symposium on Mol. Phys., Nikko, Japan (1953), pp. 65–67: *Chem. Abs.*, **52**, 6928 (10 May 1958)

Solvent effects on absorption spectra of dyes in solution are classified as (1) dielectric, (2) solvation, (3) association, (4) electrochemical effects, and (5) long-range intermolecular resonance. The dielectric effects are discussed in detail. C.O.C.

Sulphonation with Sulphur Trioxide
E. J. Carlson, G. Flint, E. E. Gilbert, and H. R. Nychka

Ind. Eng. Chem., **50**, 276–284 (March 1958)

As a result of pilot-plant studies, suitable methods are described in detail for effectively using sulphur trioxide in the sulphonation of dodecylbenzene, toluene, and petroleum lubricant raffinate, and in the sulphation of lauryl alcohol and alkylphenol–ethylene oxide condensate. W.K.R.

Sulpholysis of Sulpho Groups in Aromatic Sulphonic Acids
F. M. Vaĭnshteĭn and E. A. Shilov

J. Gen. Chem. U.S.S.R., **28**, 782–787 (March 1958)

The reaction of 2:4:6-toluenetrisulphonic acid (I) at 212°C. and of 2:4:6-phenoltrisulphonic acid (II) at 115°C. with fuming sulphuric acid, containing varying amounts of SO_3, is investigated by means of labelled ^{35}S, originally

present in one of the SO_3H groups attached to the aromatic nucleus. It is claimed that this is a two-stage reaction—(1) replacement of the SO_3H group by hydrogen (sulpholysis), followed by (2) sulphonation. For I, stage (1) is retarded whereas stage (2) is accelerated on increasing the SO_3 concn. For II, the reaction rate is independent of SO_3 concn. In the reaction with I, sulphuric acid is the SO_3 acceptor, pyrosulphuric acid being formed; in the other case, II itself acts as acceptor. In 91% aqueous H_2SO_4 the addition of K_2SO_4 facilitates the exchange of the sulpho groups in I. G.J.K.

Isolation of 2-Nitrophenol from a mixture of Mononitrophenols
T. Széll and Á. Furka

Nature, **181**, 481–482 (15 Feb. 1958)

The low solubility in dry ethanol of the sodium salt of 2-nitrophenol as compared with the 3- and 4-isomers may be used to separate the 2-isomer from mixtures; e.g. addition of sodium ethoxide to an alcoholic soln. of mononitrophenols causes pptn. of sodium 2-nitrophenoxide. A.J.

Intramolecular Hydrogen Bonding in o-Nitroanilines
L. K. Dyall and A. N. Hambly

Chem. and Ind., 262–263 (1 March 1958)

Although evidence has been given for hydrogen-bond chelation in 2-nitroacetanilides and in 1:2- and 2:1-nitronaphthylamines, the infrared spectra of *o*-nitroaniline and eight of its deriv. afford no evidence for appreciable hydrogen bonding unless nitro groups are present in both the 2- and 6-positions to the amino group. In all other cases, the combination of inductive and mesomeric effects of the nitro group shortens the N–H bond and raises the N–H stretching frequency, so that the mean absorption frequency in dil. soln. in a non-polar solvent occurs not only at a frequency higher than in aniline but close to those of *m*- and *p*-nitroanilines. H.H.H.

Derivatives of 2-Nitro- and of 2:7-Dinitro-fluorene
F. Dobrescu

Studii şi Cercetări Ştiinţifice, Timişoara, Seria Ştiinţe Chimice, 3, (3–4), 45–65 (July–Dec. 1956)

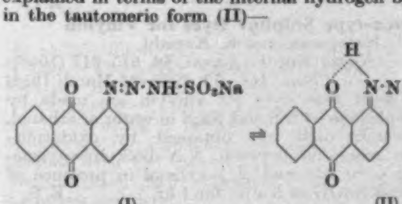
The analogy between fluorene and stilbene cpd. suggests that isomerism should be exhibited by many fluorene cpd., which would explain the differences which exist in the literature between descriptions of the same cpd. synthesised variously by different authors. This viewpoint has now been confirmed by the synthesis of 20 such cpd., and the isomeric forms, which differ in m.p., colour, and sometimes even colour reactions, are found to depend on the working conditions and the nature of the solvents employed. H.H.H.

Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives
D. Z. Zavel'skii and L. A. Lishnevskaya

VII—Reaction of Diazo Compounds with Sulphamic Acid

J. Gen. Chem. U.S.S.R., **28**, 745–755 (March 1958)

The mechanism of the reaction of aryldiazonium compounds with sulphamic acid involves the formation of the aryltriazen-*N*-sulphonic acid (I), tautomerism (prototropic) followed by decomposition into the arylamine, N_2, and H_2SO_4. Support for this mechanism is obtained from 1-anthraquinonyltriazen-3-sulphonate, which decomposes to give 1-aminoanthraquinone. The stability of 1-anthraquinonyltriazen-*N*-sulphonate is explained in terms of the internal hydrogen bond present in the tautomeric form (II)—



(I) (II)

The methylation product of I, on decomposition, produces 1-methylaminoanthraquinone, N_2, and H_2SO_4, giving support to this theory. The action of alkali upon I gives 1-anthraquinonyl azide and sulphuric acid, which is explained on the basis of a nucleophilic attack by OH^-.

Compound I is capable of existing in both red and yellow forms, the latter being more stable.

VIII—Diazonium Salts of Arylsulphamic Acids

Ibid., 755–767

With diazonium salts, arylsulphamates containing electron-accepting substituents form coloured salts, the colour ranging from yellow and red to almost black. The more electron-repelling the substituent in the aryldiazonium cation the deeper the colour of the salt formed; a similar effect is produced with the increasing electron-donating power of the substituent in the arylsulphamate anion. In water, the salts so formed, when present in high concn., are considerably more strongly coloured than in the solid state, dilution causing decolorisation. This phenomenon is explained in terms of undissociated molecules in highly concentrated solutions, dilution producing colourless ions. G.J.K.

Hydrogen Bonding Effect on the Electronic Absorption Spectrum of p-Hydroxyazobenzene
H. Baba

Bull. Chem. Soc. Japan, **31**, 169–172 (March 1958)

The changes observed (see below) when the electronic absorption spectrum of *p*-hydroxyazobenzene in *iso*-octane (I) is compared with the spectra in mixtures of I with ether and pyridine respectively are satisfactorily interpreted as due to the formation of a hydrogen bond between the solute mol. and ether or pyridine. The $\pi \rightarrow \pi^*$ bands situated in the ultraviolet region are markedly displaced to the red, while the $n \rightarrow \pi^*$ bands in the visible region are slightly displaced to the blue. H.H.H.

Azo Dyes from Aminocarbanilides
V. N. Klynev, L. A. Dogadkina, S. N. Solodushenkov, and A. A. Spryskov

J. Appl. Chem. U.S.S.R., **31**, 124–129 (Jan. 1958)

Bisdiazo compounds of substituted 4:4′-diaminocarbanilide are soluble, stable at room temp., and able to couple with Azotols at a satisfactory speed. They may be found suitable for the dyeing of cellulosic textiles by formation of the dye directly on the fibre, producing a range of colours from red to blue of satisfactory fastness to light, weathering, water, and soap. Generally speaking, the introduction of chlorine into the rings of the 4:4′-diaminocarbanilide increases the colour of the dye and decreases its fastness properties, whilst methyl and methoxy groups deepen the colour towards dark blue and blue and increase the fastness to light and weathering. The monoaminocarbanilides give similar dyes. T.Z.W.

Spectrophotometric Investigation of Sodium 1:8-Dihydroxy-2-(2-hydroxyphenylazo)-3:6 naphthalene-disulphonate (C.I. 16670) with Magnesium Ions
V. N. Tolmacher and C. G. Lomakina

Zhur. Fiz. Khim., 31, 1600–1605 (1957): *Chem. Abs.*, **52**, 5827 (10 April 1958)

Spectrophotometric studies of aqueous solutions containing Mg ions and the blue mordant dye, C.I. 16670, at equilibrium show that at pH > 10 a complex MgR_2 is formed, with an equilibrium constant $k_1 = 3{\cdot}1 \times 10^{-9}$. No definite indication of a MgR complex has been found. C.O.C.

Metal-Dye Complexes. IV—Stereochemistry of Copper(II)-Dye Complexes
H. B. Jonassen and J. R. Oliver

J. Amer. Chem. Soc., **80**, 2347–2350 (28 May 1958)

Stability, spectrophotometric and pH studies of the hydroxide, quinoline and 8-hydroxyquinoline complex compounds of Cu(II)-*oo*′-dihydroxyazobenzene, -2-carboxyphenyl-azo-β-naphthylamine and -2-carboxyphenyl-azo-naphthol complexes show that they are 1:1 addition complexes. In the 8-hydroxyquinoline complex the Cu(II) ion exhibits a coordination number of at least 5. C.O.C.

Benzidine Yellow Pigments
S. Arikawa, A. Kashioka, and T. Kawamata

Yûki Gôsei Kagaku Kyôkaishi, **16**, 29–33 (1958): *Chem. Abs.*, **52**, 6272 (25 April 1958)

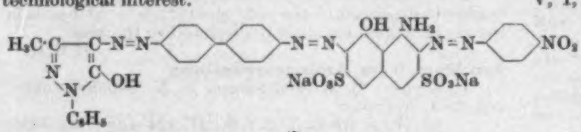
Fifteen acetoacetarylides were prepared from keten dimer and appropriate amines. They were coupled with diazotised $[3{:}4\text{-}Cl(H_2N)C_6H_3]_2$. The colour and fastness to light, heat, and oil of the resulting pigments are given. C.O.C.

Separating Component in the Synthesis of Azo Dyes
I—The Yellow Component and the Use of the Benzidine System of Azo Dyes
I. Reichel and R. Palea
Studii şi Cercetări Ştiinţifice Timişoara, Seria Ştiinţe Chimice, **3**, (1–2), 33–35 (Jan.–June 1956)

On the assumption that the benzidine system functions as the separation component in mixed disazo (or trisazo) dyes, it is found that in green disazo dyes the yellow component can be furnished by salicylic acid, phenol, 1-phenyl-3-methyl-5-pyrazolone, and possibly the anilide of acetylacetic acid, of which the most pronounced effect is shown by salicylic acid; *p*-nitroaniline is found to function as the blue component. Nine new dyes are described.

II—Direct Khaki RP, a New Benzidine-Pyrazolone Dye
I. Reichel and R. Palea
Ibid., **3**, (3–4), 9–13 (July–Dec. 1956)

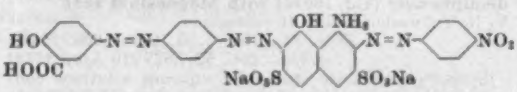
A new asymmetric benzidine dye (I), designated *Direct Khaki RP*, is prepared in the following stages—*p*-nitroaniline is diazotised and mono-coupled with H acid *ortho* to the amino group, and the resulting mono-azo dye is mono-coupled with tetrazotised benzidine, to be followed by a second coupling with 1-phenyl-3-methyl-5-pyrazolone. The tinctorial and fastness properties are described, and are such as to make the new dye of possible technological interest.



(I)

III—Phenylenediamine Systems as Separating Components
I. Reichel and A. Demian
Ibid., **3**, (3–4), 15–30 (July–Dec. 1956)

The possibility of using a tetrazotised phenylenediamine system as a separation component for the synthesis of azo dyes would appear to follow from the fact that the electronic mobilities in the aromatic nuclei of both *m*- and *p*-phenylenediamine systems are restrained, and more so in the latter than in the former. However, the experimental data so far derived from several disazo and trisazo dyes involving tetrazotised *m*- and *p*-phenylenediamine as separation components (as e.g. in II) are insufficient for definite conclusions to be drawn. Ten such new combinations are described.



(II)

IV—Modification of the Blue Component in the Synthesis of some Asymmetric Azo Dyes
I. Reichel, A. Demian, and E. Secoşan
Ibid., **4**, (1–2), 35–46 (Jan.–June 1957)

Green asymmetric disazo and trisazo dyes derived from H acid or monoazo dyes formed by coupling H acid with diazotised *p*-nitroaniline *ortho* to its amino group do not change colour appreciably when the nitro group is reduced to the amino group. Further, the colours become characteristically brighter with elimination of the amino group from the H acid, or by converting it to an azo chromophore. It thus appears that H acid or its deriv. are the essential blue components in these green asymmetric azo dyes, of which eleven new examples are described. H.H.H.

Irreversible Photobleaching of Solutions of Fluorescent Dyes. V—Photobleaching of Erythrosin (C.I. 45430)
M. Imamura
Bull. Chem. Soc. Japan, **31**, 62–66 (Jan. 1958)

The general behaviour of Erythrosin is similar to that of Eosin (C.I. 45380) (see J.S.D.C., **73**, 401 and 520 (1957)). A relatively long-lived triplet state (D') is first produced, which in aerobic aqueous soln. reacts according to—

$$D' + O_2 \rightarrow D \cdots O_2$$

and in anaerobic soln. containing alcohols according to—

$$D' + R{\cdot}OH \rightarrow DH{\bullet} + RO{\bullet}$$

$$\downarrow$$

Aldehyde.

A.J.

Fluorescent Derivatives of 1:2:3-Triazole
VI—2-Styrylnaphtho[1,2]triazolesulphonic Acids
J. Dobáš and J. Pirkl
Chem. Listy, **51**, 2330–2333 (1957): *Chem. Abs.*, **52**, 6367 (25 April 1958)

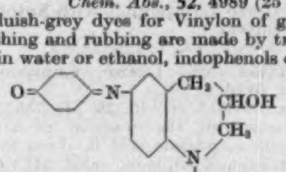
Contrary to the styryl derivatives containing in addition an auxochrome or to the bis(naphtho triazoles) based on 4:4′-diaminostilbene-2:2′-disulphonic acid which have greenish fluorescence 2-styryl-naphtho[1,2]-triazolesulphonic acids have bluish violet or greenish blue fluorescence and are good fluorescent brightening agents. Preparation of the following is described—di-Na 2-styrylnaphtho[1,2]triazole-6:8-disulphonate (I), di-Na 2-styrylnaphtho[1,2]triazole-4″:6-disulphonate (II), tri-Na 2-styrylnaphtho[1,2]triazole-2′:4″:6-trisulphonate (III), Na-2-styrylnaphtho[1,2]triazole-2′:4′:7-trisulphonate (IV) and tri-Na 2-styrylnaphtho[1,2]triazole-4″:6:8′-trisulphonate (V). II, III and IV show the best brightening effect. They all have affinity for cellulose from neutral or weakly alkaline liquors and for wool and nitrogenous fibres from weakly acid baths. They have adequate fastness to light, their fastness increasing in the order V, I, II and III. Disulphonic acids have greater substantivity than the trisulphonic acids: introducing a sulphonic group into the 2′ position decreases the substantivity considerably more than if it is introduced into the 4″-position. C.O.C.

Sulphur Dyes. XXXV—Paper Electrophoresis of Commercial Indone Direct Dyes
H. Hiyama and S. Kitahara
Kagaku to Kôgyô, **32**, 11–13 (1958): *Chem. Abs.*, **52**, 6797 (25 April 1958)

The components of commercial indone leuco dyes already known to be a mixture of reddish and bluish dyes were separated by paper electrophoresis by running 0·05 g. dye in 2 c.c. of a mixed solvent (11·7 c.c. $NH_2(CH_2)_2OH$, 5·0 c.c. 50% aq. Na_2S and 2·5 c.c. pure pyridine). Two main violet and blue spots and two secondary sky-blue and grey spots were obtained. The blue spot was absent in Direct Indone Violet B but very marked in the cases of the BB and TEB brands; it indicates presence of polysulphonamide linkages. C.O.C.

Sulphur Dyes from Tetrahydro-1-alkyl-3-quinolinol
N. Kuroki, A. Katayama, and K. Konishi
Kôgyô Kagaku Zasshi, **59**, 617–619 (1956): *Chem. Abs.*, **52**, 4989 (25 March 1958)

Blue to bluish-grey dyes for Vinylon of good fastness to light, washing and rubbing are made by treating, with S and Na_2S in water or ethanol, indophenols of formula—



(R = methyl, ethyl, butyl or 2-hydroxyethyl group). The indophenols are prepared from the reaction of tetrahydro-1-alkyl-3-quinolinols (10) with *p*-aminophenol (5·3–6·6) in presence of $K_2Cr_2O_7$ (9–12·5) and H_2SO_4 at 0–5°c. E.T.

Quinoneimine-type Sulphur Dyes for Vinylon
N. Kuroki, A. Katayama, and K. Konishi
Kôgyô Kagaku Zasshi, **59**, 615–617 (1956): *Chem. Abs.*, **52**, 4989 (25 March 1958)

Green to dark blue dyes for vinylon are made by treating indophenols with S and Na_2S in water or ethanol. The indophenols used are obtained by oxidation–condensation reactions between *N,N*-dialkylphenylenediamine and *o*- or *m*-cresol or *p*-xylenol in presence of $K_3Fe(CN)_6$ and NaOH at 0–5°c. for 1 hr. E.T.

Percyano-olefins—Tetracyanoethylene for producing Tricyanovinyl Dyes
W. J. Middleton and E. L. Little
Chem. Eng. News, **50**, 51 (28 April 1958)

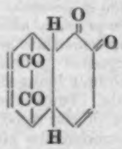
Tetracyanoethylene is the first member to be produced of a new chemical class—the percyano-olefins. Several

methods are available for its production. The only method so far studied in detail is to brominate malononitrile and then debrominate with Cu powder in boiling benzene. Tetracyanoethylene does not decompose at ≮ 600°c., is exceptionally reactive and through new and unusual addition, replacement, and cyclisation reactions will make many compounds containing cyano groups. These cyano derivatives are highly acidic, intensely coloured and easily converted into heterocyclic compounds. Thus tetracyanoethylene reacts with aromatic amines to produce a new class of brilliant orange to blue dyes— tricyanovinyl dyes. These dyes have affinity for polyester and other hydrophobic fibres. C.O.C.

First-order Calculation of Factor Group Splittings in the Electronic Spectra of Durene, Ovalene, and Phthalocyanine Crystals
L. E. Lyons
J.C.S., 1347–1351 (April 1958)

Dimer of *o*-Benzoquinone
J. Harley-Mason and A. H. Laird
J.C.S., 1718–1719 (April 1958)

Oxidation of catechol in ethereal soln. with Ag_2O gives *o*-benzoquinone (I), but in acetone soln., although I is first formed (and can be isolated by rapid evaporation of the solvent), a yellow cryst. dimer (II) soon separates and, in contrast to I, is stable indefinitely in the solid state. With *o*-phenylenediamine, II gives a quinoxaline deriv. which affords an intense green ferric chloride reaction. On spectroscopic evidence the structure proposed for II is—



H.H.H.

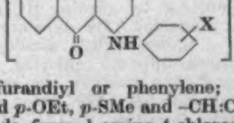
Some Applications of Raney Nickel
K. Venkataraman
J. Indian Chem. Soc., **35**, 1–18 (Jan. 1958)

A review, followed by a brief description of a general method for removing phenolic hydroxyl groups by treating the sodium sulphate half-ester with Raney nickel. Reduction of quinones, *via* the leuco forms, is accompanied by partial hydrogenation of the aromatic system. Subsequent Se-dehydrogenation is necessary to regenerate the parent hydrocarbon. E.V.T.

Anthraquinone Acid and Vat Dyes
A. Tundo

VI and VII—Acylaminoanthraquinone Derivatives of 2:5-Furandicarboxylic and Terephthalic Acids
Boll. sci. fac. chim. ind. Bologna, **15**, 75–77 and 78–79 (1957):
Chem. Abs., **52**, 4183–4184 (10 March 1958)

Blue to green-grey vat dyes for rayon are of formula—

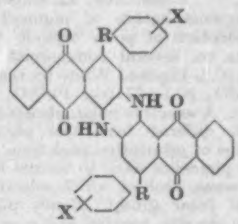


(Ar = 2:5-difurandiyl or phenylene; X = H, *o*- and *p*-OMe, *o*- and *p*-OEt, *p*-SMe and –CH:CH·CH:CH–). The dyes are made from 1-amino-4-chloroanthraquinone by (1) condensing with an appropriately substituted arylamine e.g. by refluxing in butanol with additions of Cu and K acetates and Cu powder and (2) condensing this intermediate product with the dicarboxylic acid chloride in boiling chlorobenzene. M.p. of products and light and washing fastnesses are given.

The 2:2′-disulphonic acids of the above compounds (X = H, *o*-, *m*-, *p*-Me, *p*-Cl, *p*-OMe or *p*-OEt) are made from 1-amino-4-arylaminoanthraquinone-2-sulphonic acids by refluxing with the dicarboxylic acid chloride in glacial acetic acid. The products are blue dyes for wool and silk.

VIII—Aryloxy-, Arylthio-, and Arylsulphonylindanthrone Derivatives
Ibid., 80–83

Blue vat dyes for cotton, rayon and nylon are—



(R = S, O or SO_2; X = H, *p*-Cl, *o*-, *m*-, *p*-Me, *o*-, *p*-OMe). They are obtained by the auto-condensation of certain 4-aryloxy, 4-arylthio and 4-arylsulphonyl-2-bromo-anthraquinones. These intermediates are made by refluxing (4–5 hr.) a mixture containing Na dissolved in methanol; a phenol, thiophenol or benzenesulphinic acid; *iso*-amyl alcohol and 1-amino-2:4-dibromoanthraquinone; then cooling, filtering and washing. Autocondensation occurs in boiling nitrobenzene (4–5 hr.) containing the intermediate; anhydrous K acetate and $CuSO_4$ and Cu powder.

IX—Sulphonated *NN′*-Dimethyl-4:4′-arylaminoindanthrones
Ibid., 84–86

Green acid dyes for wool and silk are made by the autocondensation of 1-methylamino-2-bromo-4-arylaminoanthraquinones. Details for making these intermediates are given. The condensation products are sulphonated with 100% H_2SO_4 containing H_3BO_3 for 10 hr. on a water-bath. Dry-cleaning fastnesses of products are tabulated. E.T.

Condensation of 8-Amino-1-aza-anthraquinone with 2-Halogenoanthraquinone
M. Yokote
Kōgyō Kagaku Zasshi, **59**, 862–864 (1956):
Chem. Abs., **52**, 6795 (25 April 1958)

8-Amino-1-aza-anthraquinone (I) boiled with 5 mol. of 2-bromoanthraquinone in presence of CuO (0·4 mol.) and K_2CO_3 (3 mol.) in nitrobenzene for 15 hr. yields a dye (II) (18% yield) which is soluble in dichlorobenzene and insoluble in acetic acid. This dye is the 8-aza analogue of Indanthren Orange 6RTK (III) (C.I. 65015). A grey compound, likewise a condensate of I, is also produced. II is a brown vat dye which yields a blue solution in conc. H_2SO_4. The main absorption band of its dyeing on cotton is 591 mμ and the dyeings have good fastness to light. Replacing the 2-bromoanthraquinone by 2-iodoanthraquinone resulted in a 10% yield of the same dye but use of 2-chloroanthraquinone yielded only the grey condensate of I. C.O.C.

Synthesis of the Pink Vat Dye 5:8-Dibenzamido-1-aza-anthraquinone
M. Yokote, T. Okawa, and K. Suzuki
Kōgyō Kagaku Zasshi, **59**, 865–866 (1956):
Chem. Abs., **52**, 6797 (28 April 1958)

8-Amino-1-aza-anthraquinone (1 mol.) was heated with oxalic acid (8–10 mol.) at 115–120°c. for 90–150 min. to give a derivative of m.p. 224°c. This was then nitrated (with KNO_3 and H_2SO_4 at 0°c.), reduced, hydrolysed (with Na_2S at the boil) to yield 5:8-diamino-1-aza-anthraquinone. This (1 mol.) was heated with dichlorobenzene (2·0–2·7 mol.) at 140–150°c. for 1–2 hr. in nitrobenzene in presence of pyridine to yield the pink vat dye 5:8-dibenzamido-aza-anthraquinone. This dye gives red with conc. H_2SO_4. Adsorption and reflection spectra of its dyeings on cotton are compared with those of Indanthren Red 5GK (C.I. 61650). C.O.C.

Spanish Red—Thiery de Menonville's *Voyage à Guaxaca*
S. M. Edelstein
Amer. Dyestuff Rep., **47**, 1–8 (13 Jan. 1958)

A picturesque account, well documented and with a map and diagrams, of the theft by Thiery de Menonville of living cochineal insects and the plants on which they fed, which he took from Spanish Mexico to French Haiti. P.T.S.

Adsorption and Surface Characteristics of some Inorganic Pigments
L. Dintenfass

Kolloid Z., **155**, 121–123 (1957):
Chem. Abs., **52**, 6808 (25 April 1958)

The heterogeneous nature of pigment surfaces was studied by adsorption of polar organic molecules from organic solvents on several commercial pigments and extenders (TiO_2 (C.I. Pigment White 6), talc (C.I. Pigment White 26), $BaSO_4$ (C.I. 77120), $PbCrO_4$ (C.I. Pigment Yellow 34), etc.). A selective polar adsorption pattern was observed which differed from the physical capillary condensation type of adsorption and from chemisorption. The surfaces of pigments seem to consist of a number of distinct active areas, each of which selectively adsorbs a specific type of polar group. Every pigment can be characterised by the size, number and type of these areas, which seem to depend more on the origin of the pigment than on its chemical structure. C.O.C.

Spectrophotometric Analysis of Compounds formed by Molybdic Acid with Complex Cyanides. I—Complexes with Prussian Compounds
S. Witekowa

Zesz. Nauk. Politech. Łódz, (18) (*Chemia* 6), 23–37 (1957)

The complexes resulting from the reaction of $(NH_4)_2MoO_4$ with $K_4[Fe(CN)_6]$, $K_3[Fe(CN)_5H_2O]$, $K_3[Fe(CN)_5NH_3]$ and $K_4[Fe(CN)_5NO_2]$ show great similarity in their light-absorption curves and the formation of only one compound at pH 4·5–5·5. The increase of the dipole moment of the cyanide by the introduction of H_2O and NH_3 into $K_4[Fe(CN)_6]$ increases the coefficient of light absorption and deepens the colour of its complex formed with $(NH_4)_2MoO_4$. The reaction between acid soln. of $(NH_4)_2MoO_4$ and the complex cyanide is possible only when the latter possesses a central ion, which, by the loss of an electron, can form a compound of similar structure at a higher level of oxidation. The cyanide, being an electron donor, polarises, similarly affecting H_2MoO_4. This mutual polarisation causes certain atoms in H_2MoO_4 to assume the Mo^{5+} structure by incorporation of one of the cyanide electrons in its system. The reducing power of the complex cyanide is too small, however, to counteract the binding energy of the two dipoles. The compounds so formed therefore have internal redox structures. T.Z.W.

PATENTS

Anthraquinone
Koppers Co. *BP* 796,395

Molten anthracene or anthracene dissolved in an inert solvent is oxidised at 150–220°C. by 98% nitric acid to give anthraquinone of 98–99% purity in good yield. Thus, anthracene of 95·8% purity (25) and nitrobenzene (200) are stirred together at 195–200°C. and a mixture of 98% HNO_3 (35) and nitrobenzene (50) is added over 1 hr. Anthraquinone crystallises on cooling the melt to 20°C. and is recovered by filtration. The cake is washed with nitrobenzene (25) and the residual nitrobenzene is then removed by steam distillation. Hot water washing and drying gives anthraquinone (25) of 99·2% purity m.p. 285–286°C. E.T.

Synthesis of Carotenoids—Food Dyes
F. Hoffmann-La Roche & Co. *BP* 796,757

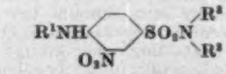
Acetylene is bilaterally condensed in an organo-metallic reaction with a C_{19}-aldehyde of formula—

$$RH_nC{\cdot}CH{:}C(CH_3)CH{:}CH{\cdot}CH{:}C(CH_3)CHO$$

(n = 1 or 2; R = a 2:6:6-trimethyl-hex[en]yl or -hex[en]-ylidene radical). When the acetylene condenses with 2 mol. of the aldehyde then a symmetrical C_{40}-(acetylenic diol) is obtained which on treatment with excess lithium aluminium hydride gives a symmetrical carotenoid. When only 1 mol. of the aldehyde takes part in the condensation then a C_{21}-(acetylenic carbinol) is formed which treated with another mol. of C_{19}-aldehyde yields a C_{40}-(acetylenic diol) which on treatment with excess lithium aluminium hydride yields the corresponding symmetrical or unsymmetrical carotenoid. The products are used as food dyes. Syntheses by these methods are described of 7:7′-dihydro-β-carotene, 3:4;3′:4′-bisdehydro-7:7′-dihydro-β-carotene, 4:4′-dehydro-β-carotene and asymmetric *iso*carotene. C.O.C.

Nitrodiarylamine Disperse Dyes
Celanese Corpn. of America *BP* 794,178

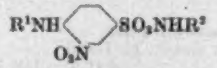
Yellow dyes for cellulose acetate having good wash fastness are—



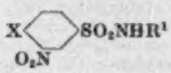
(R^1, R^2 = monoacyclic aryl hydrocarbon radicals; R^3 = hydroxyalkyl radical of 1–4 C). They are made by reacting 4-chloro-3-nitrobenzenesulphonyl chloride with *N*-hydroxyalkylanilines, e.g. phenylethanolamine, and then condensing the product with an aromatic amine containing no hydroxyalkyl group. E.T.

Nitroarylamine Disperse Dyes
Celanese Corpn. of America *BP* 794,177

Yellow dyes with good affinity for cellulose acetate and nylon and possessing good fastness to washing are—



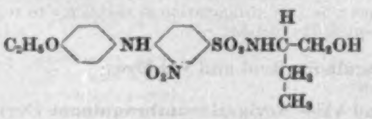
(R^1, R^2 = Ar, one of which has a substituent –COOH group; both may also have OAlk, Hal, acylamino, OH, acyl, hydroxyalkyl or hydroxyalkoxy substituents). Intermediates for these dyes are—



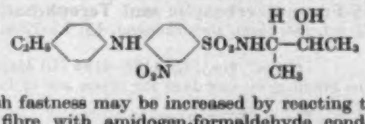
(X = Hal) prepared e.g. by reacting a 4-chloro-3-nitrobenzenesulphonyl halide in presence of an aq. acid-binding agent with an aromatic primary amine or aminocarboxylic acid. The separated intermediate is then condensed in aq. Na_2CO_3 with an aromatic primary amine or aminocarboxylic acid the product being precipitated by cooling, diluting and acidifying. Improved washing fastness is achieved by treatment of the dyed fibres with a cationic amine-formaldehyde condensate containing at least one >N–C(N–)(N–) group in addition to the N-hydroxymethyl groups which are preferably etherified. E.T.

Nitro-diphenylamine Disperse Dyes
Celanese Corpn. of America *BP* 794,176

Orange-yellow dyes for cellulose acetate rayon having good building-up properties, good light fastness and fairly good wash fastness are—



and



The wash fastness may be increased by reacting the dyes on the fibre with amidogen-formaldehyde condensates, i.e. substances containing $-\underset{|}{N}-CH_2OH$ groups. Such condensates are made e.g. by reacting HCHO with urea, or a cyclic urea or a triazine. The dyes are produced by condensing, at 50°C. under slightly alkaline conditions, 4-chloro-3-nitrobenzenesulphonyl chloride and e.g. 3-amino-2-butanol and then reacting the separated solid compound with p-phenetidine. The latter reaction takes place during 15–20 hr. in hot aq. Na_2CO_3 solution. E.T.

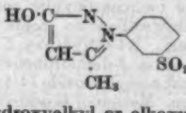
Red Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.
S *BP* 795,417

The 1:2 chromium-dye complexes of the monoazo compounds obtained by coupling diazotised 2-amino-4-chloro(or methyl)-6-nitrophenol with the pyrazolone—

dye wool, nylon, etc. red from neutral or weakly acid baths and may also be used for colouring lacquers or the spinning solutions of fibres such as cellulose acetate spun from organic solvents.

BP 795,418

Similar dyes are made by using, instead of the pyrazolone of the previous abstract, compounds of general formula—

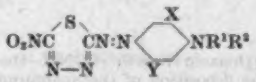


(R^1, R^2 = hydroxyalkyl or alkoxyalkyl, where the alkyl group is of < 5 C and the alkoxy of < 4 C) as coupling components. Thus 2-amino-4-chloro-6-nitrophenol is diazotised and coupled with an alkaline soln. of 3-methyl-1-phenyl-5-pyrazolone-3′-sulphonbis(β-hydroxyethyl)-amide. The monoazo compound so formed is heated in formamide at 95°C. for 8 hr. with ammonium chromium sulphate and sodium acetate to give the metal-complex, which dyes wool bluish red. E.S.

2-Amino-5-nitro-1:3:4-thiadiazole Azo Disperse Dyes

Eastman Kodak Co. *USP* 2,790,791

Compounds of formula—



(R^1 = Alk of 1–4 C, hydroxyalkyl of 2–4 C, alkoxyalkyl of 3–4 C, β-cyanoethyl, $CH_2CH_2COOCH_3$, CH_2CH_2-$COOC_2H_5$, 2:2-difluoroethyl, 2:2-difluoro-*n*-propyl, 3:3-difluoropropyl, 3:3-difluoro-*n*-butyl, 2:2:2-trifluoroethyl, or 3:3:3-trifluoropropyl; R^2 = hydroxyalkyl of 2–4 C or acetoxyethyl; X = H, methoxy or ethoxy; Y = H, CH_3, Br, Cl, methoxy, ethoxy, acetylamino, propionylamino or butyrylamino) are orange to red disperse dyes having good fastness to light and gas fume fading on cellulose acetate. They are obtained from 2-amino-5-nitro-1:3:4-thiadiazole and an appropriate coupling agent. Thus using *NN*-di-β-hydroxyethyl-*m*-toluidine as the coupling agent, a product which dyes acetate rayon salmon pink with excellent fastness to light and gas fading is obtained. C.O.C.

Red Monoazo Pigments

Sherwin-Williams Co. *USP* 2,796,415

The alkali and alkaline earth metal (esp. Ba) salts of the monoazo compound 5-amino-2-chloro*iso*propylbenzene-4-sulphonic acid→β-naphthol are faster to light than the *n*-propyl isomers, and are more transparent and of stronger yellow undertone than the homologues with methyl or ethyl groups instead of the *iso*propyl group. E.S.

Metal(Chromium)-Complex Monoazo Compounds—Pigments, Wool Dyes, and Dyes for Organic Solvents etc.

BASF *BP* 794,473

The chromium complexes of monoazo compounds obtained by coupling a diazotised *o*-alkoxyaniline with an *ortho*-coupling chlorophenol, are brighter and faster than similar dyes in which *p*-toluidine or *p*-*tert*.-butylphenol is the coupling component. Thus 2-aminoanisole-4-sulphondiethylamide is diazotised and coupled with 2:4-dichlorophenol. The monoazo compound so formed is heated at 125–130°C. with $CrCl_3$ in ethylene glycol to give the metal complex, which is reacted with salicylic acid-5-sulphonanilide by the method of *BP* 692,073 (J.S.D.C., **69**, 306 (1953)) to give a bordeaux dye for wool. E.S.

Metal(Cobalt and Chromium)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

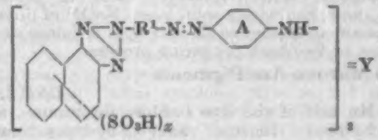
Ciba *USP* 2,784,179

The 1:2 Cr and Co complexes of monoazo dyes *o*-aminophenolsulphonamide → 3-methyl-1-phenyl-5-pyrazolone, containing an alkyl group or preferably a halogen atom in one or both of the benzene ring systems, but no COOH or SO_3H groups, are orange, red, and brown neutral-dyeing dyes for wool, nylon, etc. Thus the Cr complex of the monoazo compound 2-aminophenol-4-sulphonamide→1-*p*-chlorophenyl-3-methyl-5-pyrazolone is an orange. E.S.

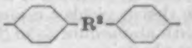
Yellow, Orange, and Brown Disazo Direct Dyes having Triazole Rings

Althouse Chemical Co. *USP* 2,795,577–8

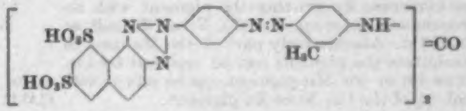
Dyes of the type—



(R^1 = *p*-phenylene, *pp*′-diphenyl, or



where R^2 is a link such as CH:CH, NH·CO, or NH·CO·NH; common substituents may be present in R^1 and A; x = integer < 4; Y = linking group such as CO or a cyanuric ring) have good light fastness and stability to resin after treatments on cellulose. Thus the monoazo compound *p*-nitroaniline→1-naphthylamine-3:6:8-trisulphonic acid is oxidised to the triazole, and the nitro group then reduced to NH_2. Diazotisation and coupling with *m*-toluidine, followed by phosgenation yields the yellow direct dye—



E.S.

Red Disazo Dye for Leather

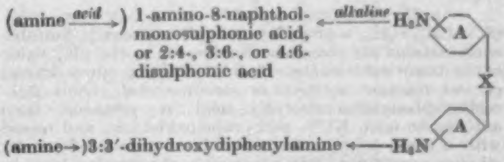
BASF *USP* 2,791,580

The disazo dye from 1 mol. of tetrazotised bis(4-amino-2-methylphenyl)methane and 1 mol. each of Schaeffer acid and R acid has good solubility and gives full, bright reds on leather. E.S.

Violet, Dark Green, Dark Blue, and Black Polyazo Dyes for Leather

FH *BP* 795,232

These dyes correspond to the general constitution—

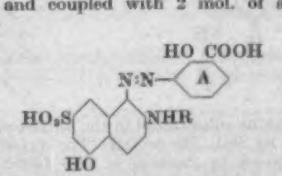


(X = direct link, CH_2:CH_2, NH·CO·NH, CO, O, S, NH, or CONH; the nuclei A may contain substituents other than OH; the brackets signify that these coupling processes are optional). Variations in the order of carrying out the coupling process are available. Thus tetrazotised benzidine is coupled in presence of aq. NaOH with 1 mol. of the acid-coupled monoazo compound sulphanilic acid→H acid, and then with 1 mol. of 3:3′-dihydroxydiphenylamine to give a bluish black resistant to acids, alkalis, and formaldehyde. E.S.

Navy Blue and Black Trisazo and Tetrakisazo Direct Dyes for Aftercoppering

S *BP* 794,366

A benzidine derivative having OH, OCH_3, OC_2H_5, OCH_2COOH or COOH groups in the 3:3′-positions is tetrazotised and coupled with 2 mol. of an aminoazo compound—



(R = Alk, hydroxyalkyl, alkoxyalkyl, *cyclo*alkyl, aralkyl, or Ar; the ring A may contain substituents e.g. NO_2, Cl, CH_3·SO_2, SO_3H, SO_2·NH_2, etc.), to give the title dyes. Or 1 mol. of such an aminoazo compound and 1 mol. of any

coupling component having a chelatable group, e.g. salicylic acid, an *o*-coupling naphthol or pyrazolone, may be used. Thus tetrazotised dianisidine is coupled with 1 mol. of the acid-coupled monoazo compound 3-amino-5-sulphosalicylic acid→*N*-phenyl-J acid in presence of Na_2CO_3, and then with a soln. in aq. NaOH of β-naphthol. The trisazo compound so formed dyes cellulose navy blue by a one- or two-bath coppering process. E.S.

Red to Maroon Azo Pigments
DuP *USP* 2,649,383

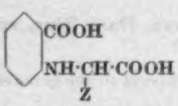
The Mn salt of the dye 4-chloro (or bromo-, iodo- or nitro-)-2-amino benzoic acid→3-hydroxy-2-naphthoic acid has excellent fastness to weathering when used in coating compositions. In addition it shows a high degree of transparency so that it yields a desirable "two-tone" or flash when used with aluminium flake (C.I. Pigment Metal 1) in finishes for car bodies.

USP 2,791,513

When the above pigment is used in short oil baking enamels containing resins of the urea-formaldehyde type it shows tendency toward reactivity, i.e. there is progressive and marked increase in viscosity with time of storage so that ultimately a gel structure is set up which is no longer of use as an enamel. When used in cellulose nitrate lacquers it is markedly sensitive to small amounts of water. These two defects are overcome by treating the pigment with an ammoniacal solution of a Cu, Ni or Zn salt at 50–100°c. Alternatively part of the Mn used to precipitate the pigment can be replaced by Cu, Ni or Zn or the Mn pigment can be mixed with up to 20 mol. % of the Cu, Ni or Zn pigment. C.O.C.

Azoic Dyes—Use of α-(*o*-Carboxyphenylamino)-aliphatic Acids in Preparing Triazenes
Ciba *BP* 795,339

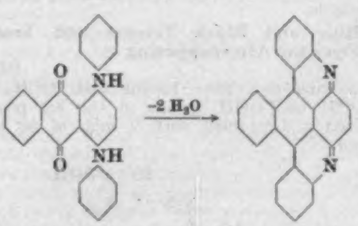
Triazenes suitable for azoic dyeing and printing processes are made by condensing suitable diazo compounds with dicarboxylic acids—



(Z = CH_3, C_2H_5, *n*-propyl, *iso*propyl, or phenyl). Suitable combinations are chosen with reference to the pK_A value of the diazotisable amine. Rapid drying (e.g. spray drying) of the reaction solution is recommended. Thus β-(*o*-carboxyphenylamino)butyric acid is prepared from anthranilic acid, KCN, and propionaldehyde, and mixed with a diazo solution from 4-amino-3-nitroanisole under alkaline conditions. After screening, the solution is spray dried to give an 80–85% yield of the triazene. E.S.

Dicoeramidonines—Dyes for Acrylic Fibres
FBy *BP* 794,807

Olive-green to grey dyes of excellent light fastness on polymers or copolymers of acrylonitrile are mono-quaternary salts of dicoeramidonines. The intermediates for these dyes are made by double ring-closures, e.g. by means of H_2SO_4, of 1:4- or 1:5-dianilidoanthraquinones. Thus—



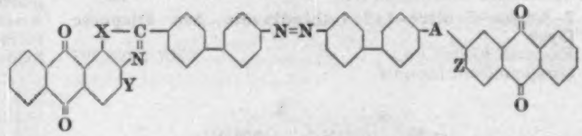
They may also be substituted in the anthraquinone and/or aryl nucleus by Hal, Alk or OH. The dicoeramidonine is then quaternised by heating at 100–150°c. in an inert solvent, e.g. *o*-dichlorobenzene or nitrobenzene, with dimethyl sulphate. Only the mono-quaternary compound is formed which crystallises when the melt is cooled. The products are water-soluble and are dyed from aq. dyebaths containing Na acetate and glacial acetic acid. E.T.

Polyhydroxy-β-Acylaminoanthraquinone Disperse Dyes
Ciba *USP* 2,819,288

Red dyes for polyester fibres, e.g. Terylene, are made by acylating polyhydroxy-β-aminoanthraquinones. The anthraquinone nucleus has, preferably, 2–4 α-hydroxy groups, at least one β-amino group and no water-solubilising groups although it may have further simple substituents. Aliphatic monocarboxylic acids of 2–12 C and benzoic or hexahydro-benzoic acids are suitable acylating agents. Thus, 1:4-dihydroxy-2-aminoanthraquinone (12·7) is heated in *o*-dichlorobenzene (36) with dimethylaniline (6·6) and acetylchloride (4·4) until there is no further colour change. Methanol is added to the cooled reaction mixture and the precipitated dye is filtered and washed with methanol. The product is dissolved in 90–95% H_2SO_4 at −10°c., poured on to ice, filtered and washed. A dispersion of the dye colours Terylene a fast red-orange. E.T.

Sulphonated Vat Dyes
DuP *USP* 2,785,156

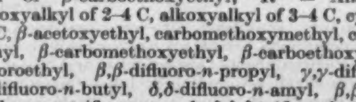
Yellow vat dyes of formula—



(A = oxazole, thiazole rings or –CONH– the N atom being attached to the β-position of the anthraquinone nucleus; X = O or S; Y and/or Z = H) are sulphonated so that they contain 0·9 to 1·5 SO_3H groups. The products, which are still water-insoluble, have much improved printing properties but maintain the excellent wash-fastness of the original dyes. A method of making one of the un-sulphonated vat dyes is given and this is then sulphonated as follows. The dye (20) is dissolved in 25% oleum (100) below 60°c. The solution is heated at 105–108°c. for 40 min., cooled to 50°c. and drowned into hot NaCl (150) in water (1500). The insoluble product is filtered, washed acid-free with warm water and purified by bleaching with NaOCl in the presence of Na_2CO_3. E.T.

Benzothiazoleazo-aniline Disperse Dyes
Eastman Kodak *USP* 2,785,157

Bright scarlet, red, pink, violet and blue dyes for cellulose acetate of excellent fastness to light, gas-fumes and sublimation are of formula—



(R^1 = H, Alk of 1–4 C, hydroxyalkyl of 2–4 C, alkoxy-alkyl of 3–4 C, cyanoalkyl of 3–5 C, β-acetoxyethyl, carbomethoxymethyl, carboethoxymethyl, β-carbomethoxyethyl or β-carboethoxyethyl; R^2 = Alk of 1–4 C, hydroxyalkyl of 2–4 C, alkoxyalkyl of 3–4 C, cyanoalkyl of 3–5 C, β-acetoxyethyl, carbomethoxymethyl, carboethoxymethyl, β-carbomethoxyethyl, β-carboethoxyethyl, β,β-difluoroethyl, β,β-difluoro-*n*-propyl, γ,γ-difluoropropyl, γ,γ-difluoro-*n*-butyl, δ,δ-difluoro-*n*-amyl, β,β,β-trifluorobutyl, γ,γ,γ-trifluoropropyl, δ,δ,δ-trifluorobutyl; X = H, Br, Cl, CH_3, C_2H_5, CH_3O, C_2H_5O, CH_3CONH, *n*-propionylamino or *n*-butylamino; Y = H, CH_3, C_2H_5, CH_3O or C_2H_5O; Z = allyl or Alk of 1–4 C). The 2-amino-6-(alkyl or allyl) sulphonylbenzothiazole intermediates are diazotised in H_2SO_4 or mixtures of mineral and organic acids. Thus, 2-amino-6-methylsulphonylbenzothiazole (57) is dissolved in 50% H_2SO_4 (1250) at 90°c. To this solution at −10°c. is added $NaNO_2$ (17·5) in conc. H_2SO_4 (125) at < −3°c. followed by stirring at −5°c. Coupling then follows in weak H_2SO_4. Thus, the diazonium solution is stirred into *N*-β-hydroxyethyl-*N*-β-cyanoethylaniline (46·5) in 10% aq. H_2SO_4 (1250) at −5°c. The dyes obtained are dischargeable and may also be used on wool, silk, nylon, terylene and acrylonitrile fibres. E.T.

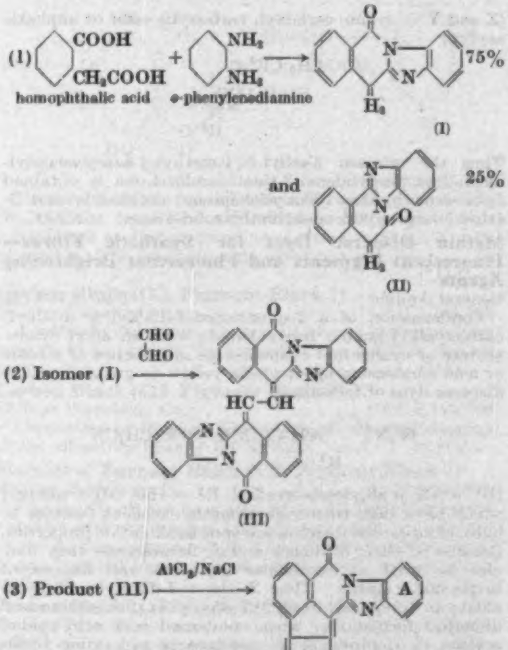
Anthraquinone Vat Dyes
Ciba *BP* 794,849

Grey vat dyes of good fastness to chlorine are produced by treating 1-aminoanthraquinones having a H atom in

the 4-position first with an oxidising agent, e.g. MnO_2, CrO_3, etc., in aq. H_2SO_4 and then with a carbazolising agent in presence of a tertiary amine. Thus, 1-aminoanthraquinone (44·6) is dissolved in 96% H_2SO_4 (816) and the solution is added to water (1184) with cooling so that the final temperature is 18°c. A suspension of pyrolusite (27), containing 82·5% MnO_2, in 40% H_2SO_4 (152 by vol.) is added and the whole is stirred for 60 hr. at 20–25°c. The product is separated by filtration, washed with 40% H_2SO_4 and then with water until neutral. The dried product (5) is added to anhydr. $AlCl_3$ (25) and pyridine (60) and the melt is heated at 145°c. to distill off the pyridine. After a further 6 hr. heating it is precipitated into 4% aq. NaOH (1000) and purified by vatting with $Na_2S_2O_4$ at 60–65°c. and air oxidation. The dyes so obtained are not chemically pure compounds and differ from those obtained by carbazolising linear polyanthrimides. It is therefore assumed that the first stage of the process, i.e. the oxidation, is more complex than the mere formation of polyanthrimides. E.T.

Acedibenzimidazoisoquinolone Vat Dyes
DuP *USP* 2,785,167

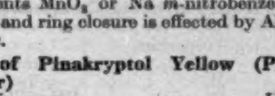
Violet vat dyes, applied to cellulosic fibres from green alkaline hydrosulphite vats, are made by the reactions—



The rings A, B may be substituted by Cl or Br. Reaction (1) occurs at high temperature in an inert solvent, e.g. boiling trichlorobenzene, the product being separated by cooling, adding ethanol and filtering. Reaction (2) employs aq. glyoxal in e.g. dimethylformamide at 100°c. Reaction (3) consists of an oxidative cyclisation; as oxidising agents MnO_2 or Na *m*-nitrobenzenesulphonate may be used and ring closure is effected by $AlCl_3$ and NaCl at 120–135°c. E.T.

Synthesis of Pinakryptol Yellow (Photographic Desensitizer)
General Aniline *USP* 2,794,802

Pinakryptol yellow—



is obtained in only 20–30% yield using the methods of *GP* 396,402 and 468,093 by condensing a 1-methyl-6-ethoxyquinaldinium salt, e.g. the methosulphate, with *m*-nitrobenzaldehyde. The present method claims that a high yield and nearly analytical purity can be obtained from the same reactants if temperature is controlled so that 80°c. is reached within 2–3 min. of bringing the reactants together. The acid liberated by the reaction is taken up by a strong base, e.g. piperidine, and a solvent, e.g. *iso*propyl alcohol, is used. After reaching 80°c. in 2–3 min. the reaction is completed by refluxing. The product is separated in 92–97% yield; m.p. 267°c. (cf. analytical sample 266–8°c.) by cooling, filtering and methanol washing. E.T.

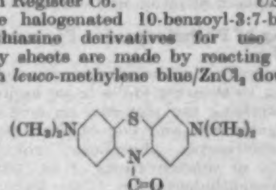
Perylene Tetracarboxylic Acid Diimide Vat Dyes
General Aniline *USP* 2,794,805

Grey vat dyes applicable to cellulose from alkaline hydrosulphite vats are—

and its nuclear halo- (Br or Cl) and amino-substituted derivatives. They are made by *either* (1) condensing a perylene-3:4:9:10-tetracarboxylic acid diimide (1 mol.) with acrylonitrile (2 mol.) in presence of an organic quaternary ammonium base e.g. trimethylphenyl ammonium hydroxide *or* (2) condensing a perylene-3:4:9:10-tetracarboxylic acid anhydride with β-aminopropionitrile with the elimination of H_2O or with imino-bis-β:β'-propionitrile with the elimination of water and acrylonitrile. E.T.

Halogen Derivatives of *leuco*-Methylene Blue
National Cash Register Co. *USP* 2,784,186

Light-stable halogenated 10-benzoyl-3:7-bis(dimethylamino)phenothiazine derivatives for use in coating manifold copy sheets are made by reacting halobenzoyl chlorides with *leuco*-methylene blue/$ZnCl_2$ double salts to give



(R = Cl or Br; *n* = 1–5). Thus, *leuco*-methylene blue/$ZnCl_2$ double salt (73) in pyridine (122) is heated at 90°c. and *o*-chlorobenzoyl chloride (50) is added dropwise. The mixture is refluxed (1–1½ hr.) and poured into water. The separated semi-solid material is mixed with NaOH (48) in water (1000), warmed 30 min. and the alkaline liquid is decanted. The solid is water-washed, dissolved in hot benzene (2000) the solution being then treated with Fuller's earth and filtered. A white crystalline solid is obtained by concentrating the solution and adding petroleum ether. E.T.

Readily Soluble Compositions containing 4-Methyl-7-aminocoumarin Fluorescent Brightening Agents
Saul & Co. *USP* 2,791,564

A mixture of a surface active substance and > 5% by wt. of 4-methyl-7-diethylaminocoumarin is readily soluble in water. It is very suitable for use as a fluorescent brightening agent to be added to washing powders. C.O.C.

Tertiary Substituted 2-Aryl Aryleneazoles—Fluorescent Brightening Agents
American Cyanamid Co. *USP* 2,793,192

Compounds of formula—

coupling component having a chelatable group, e.g. salicylic acid, an *o*-coupling naphthol or pyrazolone, may be used. Thus tetrazotised dianisidine is coupled with 1 mol. of the acid-coupled monoazo compound 3-amino-5-sulphosalicylic acid→*N*-phenyl-J acid in presence of Na_2CO_3, and then with a soln. in aq. NaOH of β-naphthol. The trisazo compound so formed dyes cellulose navy blue by a one- or two-bath coppering process. E.S.

Red to Maroon Azo Pigments

DuP *USP* 2,649,383

The Mn salt of the dye 4-chloro (or bromo-, iodo- or nitro-)-2-amino benzoic acid→3-hydroxy-2-naphthoic acid has excellent fastness to weathering when used in coating compositions. In addition it shows a high degree of transparency so that it yields a desirable "two-tone" or flash when used with aluminium flake (C.I. Pigment Metal 1) in finishes for car bodies.

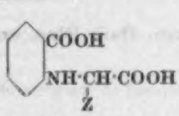
USP 2,791,513

When the above pigment is used in short oil baking enamels containing resins of the urea-formaldehyde type it shows tendency toward reactivity, i.e. there is progressive and marked increase in viscosity with time of storage so that ultimately a gel structure is set up which is no longer of use as an enamel. When used in cellulose nitrate lacquers it is markedly sensitive to small amounts of water. These two defects are overcome by treating the pigment with an ammoniacal solution of a Cu, Ni or Zn salt at 50–100°c. Alternatively part of the Mn used to precipitate the pigment can be replaced by Cu, Ni or Zn or the Mn pigment can be mixed with up to 20 mol. % of the Cu, Ni or Zn pigment. C.O.C.

Azoic Dyes—Use of α-(*o*-Carboxyphenylamino)-aliphatic Acids in Preparing Triazenes

Ciba *BP* 795,339

Triazenes suitable for azoic dyeing and printing processes are made by condensing suitable diazo compounds with dicarboxylic acids—

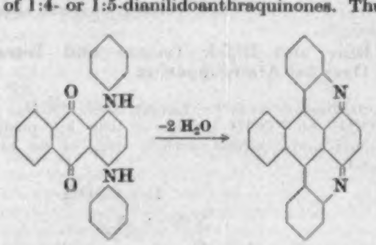


($Z = CH_3$, C_2H_5, *n*-propyl, *iso*propyl, or phenyl). Suitable combinations are chosen with reference to the pK_A value of the diazotisable amine. Rapid drying (e.g. spray drying) of the reaction solution is recommended. Thus β-(*o*-carboxyphenylamino)butyric acid is prepared from anthranilic acid, KCN, and propionaldehyde, and mixed with a diazo solution from 4-amino-3-nitroanisole under alkaline conditions. After screening, the solution is spray dried to give an 80–85% yield of the triazene. E.S.

Dicoeramidonines—Dyes for Acrylic Fibres

FBy *BP* 794,807

Olive-green to grey dyes of excellent light fastness on polymers or copolymers of acrylonitrile are mono-quaternary salts of dicoeramidonines. The intermediates for these dyes are made by double ring-closures, e.g. by means of H_2SO_4, of 1:4- or 1:5-dianilidoanthraquinones. Thus—



They may also be substituted in the anthraquinone and/or aryl nucleus by Hal, Alk or OH. The dicoeramidonine is then quaternised by heating at 100–150°c. in an inert solvent, e.g. *o*-dichlorobenzene or nitrobenzene, with dimethyl sulphate. Only the mono-quaternary compound is formed which crystallises when the melt is cooled. The products are water-soluble and are dyed from aq. dyebaths containing Na acetate and glacial acetic acid. E.T.

Polyhydroxy-β-Acylaminoanthraquinone Disperse Dyes

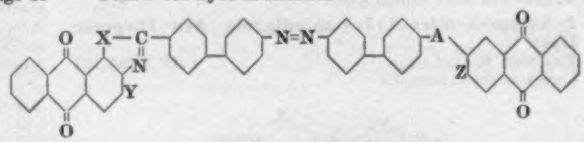
Ciba *USP* 2,819,288

Red dyes for polyester fibres, e.g. Terylene, are made by acylating polyhydroxy-β-aminoanthraquinones. The anthraquinone nucleus has, preferably, 2–4 α-hydroxy groups, at least one β-amino group and no water-solubilising groups although it may have further simple substituents. Aliphatic monocarboxylic acids of 2–12 C and benzoic or hexahydro-benzoic acids are suitable acylating agents. Thus, 1:4-dihydroxy-2-aminoanthraquinone (12·7) is heated in *o*-dichlorobenzene (36) with dimethylaniline (6·6) and acetylchloride (4·4) until there is no further colour change. Methanol is added to the cooled reaction mixture and the precipitated dye is filtered and washed with methanol. The product is dissolved in 90–95% H_2SO_4 at −10°c., poured on to ice, filtered and washed. A dispersion of the dye colours Terylene a fast red-orange. E.T.

Sulphonated Vat Dyes

DuP *USP* 2,785,156

Yellow vat dyes of formula—

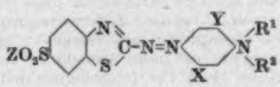


(A = oxazole, thiazole rings or –CONH– the N atom being attached to the β-position of the anthraquinone nucleus; X = O or S; Y and/or Z = H) are sulphonated so that they contain 0·9 to 1·5 SO_3H groups. The products, which are still water-insoluble, have much improved printing properties but maintain the excellent wash-fastness of the original dyes. A method of making one of the unsulphonated vat dyes is given and this is then sulphonated as follows. The dye (20) is dissolved in 25% oleum (100) below 60°c. The solution is heated at 105–108°c. for 40 min., cooled to 50°c. and drowned into hot NaCl (150) in water (1500). The insoluble product is filtered, washed acid-free with warm water and purified by bleaching with NaOCl in the presence of Na_2CO_3. E.T.

Benzothiazoleazo-aniline Disperse Dyes

Eastman Kodak *USP* 2,785,157

Bright scarlet, red, pink, violet and blue dyes for cellulose acetate of excellent fastness to light, gas-fumes and sublimation are of formula—



(R^1 = H, Alk of 1–4 C, hydroxyalkyl of 2–4 C, alkoxyalkyl of 3–4 C, cyanoalkyl of 3–5 C, β-acetoxyethyl, carbomethoxymethyl, carboethoxymethyl, β-carbomethoxyethyl or β-carboethoxyethyl; R^2 = Alk of 1–4 C, hydroxyalkyl of 2–4 C, alkoxyalkyl of 3–4 C, cyanoalkyl of 3–5 C, β-acetoxyethyl, carbomethoxymethyl, carboethoxymethyl, β-carbomethoxyethyl, β-carboethoxyethyl, β,β-difluoroethyl, β,β-difluoro-*n*-propyl, γ,γ-difluoropropyl, γ,γ-difluoro-*n*-butyl, δ,δ-difluoro-*n*-amyl, β,β,β-trifluorobutyl, γ,γ,γ-trifluoropropyl, δ,δ,δ-trifluorobutyl; X = H, Br, Cl, CH_3, C_2H_5, CH_3O, C_2H_5O, CH_3CONH, *n*-propionylamino or *n*-butylamino; Y = H, CH_3, C_2H_5, CH_3O or C_2H_5O; Z = allyl or Alk of 1–4 C). The 2-amino-6-(alkyl or allyl) sulphonylbenzothiazole intermediates are diazotised in H_2SO_4 or mixtures of mineral and organic acids. Thus, 2-amino-6-methylsulphonylbenzothiazole (57) is dissolved in 50% H_2SO_4 (1250) at 90°c. To this solution at −10°c. is added $NaNO_2$ (17·5) in conc. H_2SO_4 (125) at < -3°c. followed by stirring at −5°c. Coupling then follows in weak H_2SO_4. Thus, the diazonium solution is stirred into *N*-β-hydroxyethyl-*N*-β-cyanoethylaniline (46·5) in 10% aq. H_2SO_4 (1250) at −5°c. The dyes obtained are dischargeable and may also be used on wool, silk, nylon, terylene and acrylonitrile fibres. E.T.

Anthraquinone Vat Dyes

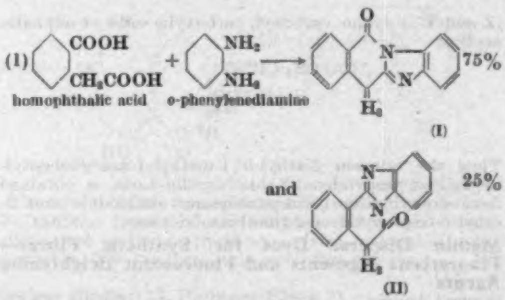
Ciba *BP* 794,849

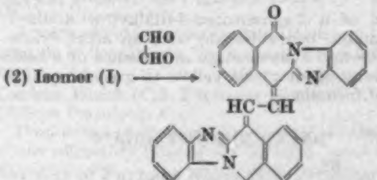
Grey vat dyes of good fastness to chlorine are produced by treating 1-aminoanthraquinones having a H atom in

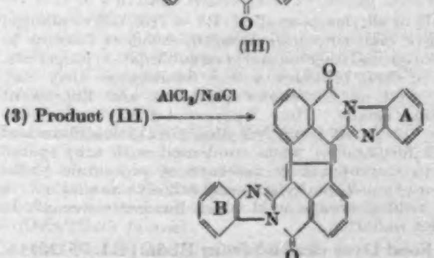
the 4-position first with an oxidising agent, e.g. MnO_2, CrO_3, etc., in aq. H_2SO_4 and then with a carbazolising agent in presence of a tertiary amine. Thus, 1-aminoanthraquinone (44·6) is dissolved in 96% H_2SO_4 (816) and the solution is added to water (1184) with cooling so that the final temperature is 18°c. A suspension of pyrolusite (27), containing 82·5% MnO_2, in 40% H_2SO_4 (152 by vol.) is added and the whole is stirred for 60 hr. at 20–25°c. The product is separated by filtration, washed with 40% H_2SO_4 and then with water until neutral. The dried product (5) is added to anhydr. $AlCl_3$ (25) and pyridine (60) and the melt is heated at 145°c. to distill off the pyridine. After a further 6 hr. heating it is precipitated into 4% aq. NaOH (1000) and purified by vatting with $Na_2S_2O_4$ at 60–65°c. and air oxidation. The dyes so obtained are not chemically pure compounds and differ from those obtained by carbazolising linear polyanthrimides. It is therefore assumed that the first stage of the process, i.e. the oxidation, is more complex than the mere formation of polyanthrimides. E.T.

Acedibenzimidazoisoquinolene Vat Dyes
DuP *USP* 2,785,167

Violet vat dyes, applied to cellulosic fibres from green alkaline hydrosulphite vats, are made by the reactions—



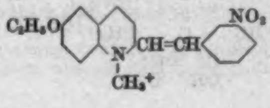




The rings A, B may be substituted by Cl or Br. Reaction (1) occurs at high temperature in an inert solvent, e.g. boiling trichlorobenzene, the product being separated by cooling, adding ethanol and filtering. Reaction (2) employs aq. glyoxal in e.g. dimethylformamide at 100°c. Reaction (3) consists of an oxidative cyclisation; as oxidising agents MnO_2 or Na *m*-nitrobenzenesulphonate may be used and ring closure is effected by $AlCl_3$ and NaCl at 120–135°c. E.T.

Synthesis of Pinakryptol Yellow (Photographic Desensitizer)
General Aniline *USP* 2,794,802

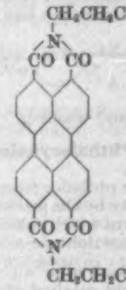
Pinakryptol yellow—



is obtained in only 20–30% yield using the methods of *GP* 396,402 and 468,093 by condensing a 1-methyl-6-ethoxyquinaldinium salt, e.g. the methosulphate, with *m*-nitrobenzaldehyde. The present method claims that a high yield and nearly analytical purity can be obtained from the same reactants if temperature is controlled so that 80°c. is reached within 2–3 min. of bringing the reactants together. The acid liberated by the reaction is taken up by a strong base, e.g. piperidine, and a solvent, e.g. *iso*propyl alcohol, is used. After reaching 80°c. in 2–3 min. the reaction is completed by refluxing. The product is separated in 92–97% yield; m.p. 267°c. (cf. analytical sample 266–8°c.) by cooling, filtering and methanol washing. E.T.

Perylene Tetracarboxylic Acid Diimide Vat Dyes
General Aniline *USP* 2,794,805

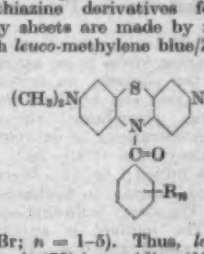
Grey vat dyes applicable to cellulose from alkaline hydrosulphite vats are—



and its nuclear halo- (Br or Cl) and amino-substituted derivatives. They are made by *either* (1) condensing a perylene-3:4:9:10-tetracarboxylic acid diimide (1 mol.) with acrylonitrile (2 mol.) in presence of an organic quaternary ammonium base e.g. trimethylphenyl ammonium hydroxide *or* (2) condensing a perylene-3:4:9:10-tetracarboxylic acid anhydride with β-aminopropionitrile with the elimination of H_2O or with imino-bis-β:β′-propionitrile with the elimination of water and acrylonitrile. E.T.

Halogen Derivatives of *leuco*-Methylene Blue
National Cash Register Co. *USP* 2,784,186

Light-stable halogenated 10-benzoyl-3:7-bis(dimethylamino)phenothiazine derivatives for use in coating manifold copy sheets are made by reacting halobenzoyl chlorides with *leuco*-methylene blue/$ZnCl_2$ double salts to give



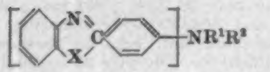
(R = Cl or Br; *n* = 1–5). Thus, *leuco*-methylene blue/$ZnCl_2$ double salt (73) in pyridine (122) is heated at 90°c. and *o*-chlorobenzoyl chloride (50) is added dropwise. The mixture is refluxed (1–1½ hr.) and poured into water. The separated semi-solid material is mixed with NaOH (48) in water (1000), warmed 30 min. and the alkaline liquid is decanted. The solid is water-washed, dissolved in hot benzene (2000) the solution being then treated with Fuller's earth and filtered. A white crystalline solid is obtained by concentrating the solution and adding petroleum ether. E.T.

Readily Soluble Compositions containing 4-Methyl-7-aminocoumarin Fluorescent Brightening Agents
Saul & Co. *USP* 2,791,564

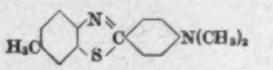
A mixture of a surface active substance and > 5% by wt. of 4-methyl-7-diethylaminocoumarin is readily soluble in water. It is very suitable for use as a fluorescent brightening agent to be added to washing powders. C.O.C.

Tertiary Substituted 2-Aryl Aryleneazoles—Fluorescent Brightening Agents
American Cyanamid Co. *USP* 2,793,192

Compounds of formula—



(X = S, NH or O; R¹ and R² = same or different aliphatic hydrocarbons which may be joined to form an alicyclic ring) are fluorescent brightening agents. When the tertiary amino nitrogen is bonded to the aryl ring then there is reddish blue fluorescence, and greenish-blue when it is bonded to the arylene azole nucleus. They have affinity for both cellulosic and non-cellulosic materials. Thus to a mixture of dehydro thio-*p*-toluidine, soda ash and methyl alcohol, methyl iodide is gradually added. The whole is then refluxed until reaction ceases, cooled and drowned in water. The crude product obtained by filtration is washed, dissolved in N-HCl at 80°C. and alcohol and activated charcoal added. The solution is clarified hot and the filtrate cooled. The precipitated amine hydrochloride is basified and the free amine which has the constitution—



is isolated by filtration and washing. C.O.C.

Chlorinated Copper Phthalocyanine
Ansbacher-Siegle Corpn. *USP* 2,793,214

Highly chlorinated Cu phthalocyanines were obtained in good yield and high purity by the method of *USP* 2,662,085 (J.S.D.C., **71**, 421 (1955)) in which chlorination, at relatively low temperature, in chlorsulphonic acid gave

$$[(C_6H_{4-n}Cl_n)C_2N_2]_4\,Cu$$

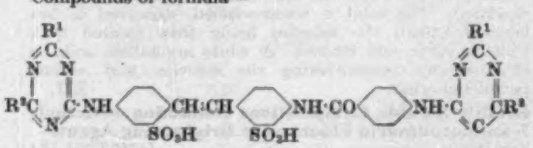
(n = 12–15). The present method gives this high degree of chlorination and the same freedom from unwanted by-products without the disadvantages and dangers involved in the use of chlorsulphonic acid. Instead of chlorsulphonic acid, organic sulphonic acids which have m.p. < 120°C., b.p. > 120°C., no oxidising properties and which are not decomposed by water are used. Thus, benzenesulphonic, benzenedisulphonic, *p*-chlorobenzene sulphonic, *m*-nitrobenzenesulphonic acids etc. may be used and are recoverable for re-use. Chlorination with Cl_2 gas at 60–120°C. with addition of a suitable catalyst (e.g. S, I, S chlorides, I chlorides etc.) is followed by drowning into water or dil. alkali, filtration and washing. E.T.

Acid Milling of Phthalocyanine Pigments
General Aniline *BP* 795,414

The transparency of phthalocyanine pigments is improved by subjecting to shearing forces in an appropriate mill a mixture comprising the pigment, an acid-stable, water-soluble inorganic salt and one of the following acids—sulphuric, chloracetic, phosphoric, chlorsulphonic or alkylsulphonic at concentrations of 68–100%. Thus, Cu hexadecachlorophthalocyanine (100), 96% H_2SO_4 (100) and Na_2SO_4 (200) are milled 1 hr. in a Werner–Pfleiderer mill and the resulting magma is discharged into water (1000) with stirring. The slurry is filtered and the product washed acid-free. The method is applicable to any phthalocyanine, e.g. metal-free, Cu, Ni, Co, Zn, Pb, halogenated phthalocyanines. E.T.

Fluorescent Brightening Agents
Hickson & Welch Ltd. *BP* 796,524

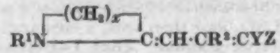
Compounds of formula—



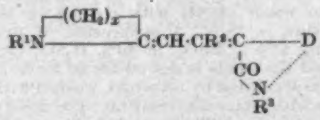
(R¹ = NH_2 or residue of a primary or secondary amine not containing an auxochrome or chromophore and which is linked to the adjacent triazine ring through its N atom; R² = Cl, OH or R¹) exhibit greenish fluorescence and are good fluorescent brightening agents for cellulosic materials. C.O.C.

Methin Dyes
Ilford *BP* 795,134

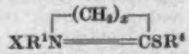
Methin dyes of formula—



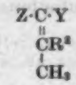
or



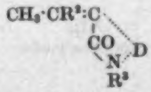
(R¹ = Alk or Ar; R² = H, Alk or alkoxy; R³ = H, Alk, aralkyl or Ar; D = atoms to complete a 5- or 6-membered ring; x = 4, 5 or 6) are obtained by condensing a compound of formula—



(R_4 = Alk or aralkyl) in presence of a basic condensing agent with a compound of formula—



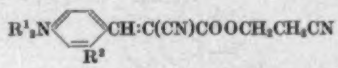
(Z and Y = cyano, carboxyl, carboxylic ester or aliphatic acyl) or



Thus the crimson 3-ethyl-5-[1-methyl-1-aza*cyclo*heptylidene-2)-*iso*propylidene]-2-thiothiazolid-4-one is obtained from 2-methylthio-1-aza*cyclo*heptene methiodide and 3-ethyl-5-*iso*propylidene-2-thiothiazolid-4-one. C.O.C.

Methin Disperse Dyes for Synthetic Fibres—Fluorescent Pigments and Fluorescent Brightening Agents
General Aniline *USP* 2,789,125

Condensation of a 2-substituted-4-dialkyl(or di[alkylcarboxyalkyl])amino benzaldehyde with an alkyl cyanoacetate or cyanoethyl cyanoacetate in presence of a basic or acid condensing agent yields yellow to greenish-yellow disperse dyes of formula—



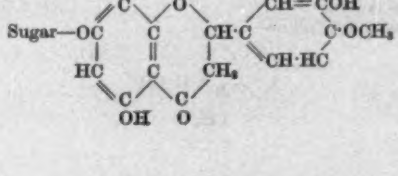
(R¹ = Alk or alkylcarboxyalkyl; R² = Hal, OH or alkoxy) which have high tinctorial strength, excellent fastness to light, chlorine and washing and good sublimation properties. Because of their brilliance and/or fluorescence they may also be used as fluorescent pigments and fluorescent brightening agents. Thus 2-ethoxy-4-diethylaminobenzaldehyde (prepared from *NN*-diethyl-*m*-phenetidine and dimethyl formamide) when condensed with ethylcyanoacetate in *iso*propanol in presence of piperidine yields ethyl α-cyano-4(diethylamino)-2-ethoxycinnamate, a brilliant reddish-orange solid which fluoresces strongly in ultraviolet radiation. C.O.C.

Yellow Food Dyes derived from Bixin (C.I. 75120)
Keystone Chemurgic Corpn. *USP* 2,793,124

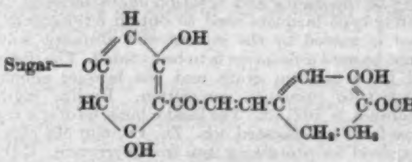
Pure, crystalline red bixin is converted into an oil-soluble yellow resinous material suitable as a food dye by heating it in a lower aliphatic monocarboxylic acid in absence of oxygen and in presence of the anhydride of the acid, e.g. acetic anhydride, as catalyst. The product is preferably used in solution in triacetin or tributyrin. C.O.C.

Wood Stains from Flavonoid Compounds
Commonwealth Engineering Co. *USP* 2,792,386

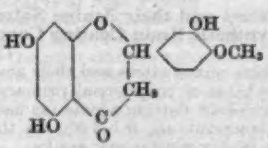
Flavonoids, e.g. hesperidin—



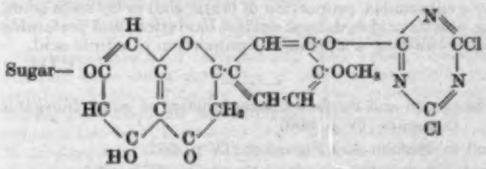
hesperidin chalcone—



or hesperitin—



are treated with cyanuric chloride in presence of $AlCl_3$ and dispersed in CS_2 to yield brown substances which are soluble in water and useful as fast-to-light wood stains. The compound formed from hesperidin probably has the following structure—



USP 2,792,387

The flavonoids are treated with $AlCl_3$ while suspended in CS_2 and the brownish coloured stain extracted with a solvent such as dioxan, pyridine or water, in which it may be soluble. The products stain wood fast-to-light. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Houdry Process Corpn. *USP* 2,794,709

A method of obtaining Carbon Black and hydrogen from gaseous hydrocarbons, particularly those found in natural gas. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *USP* 2,794,710

Production of Furnace Black from oil without slagging of the refractory liner of the furnace. C.O.C.

Slurries of Furnace Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *USP* 2,794,749

Furnace Black can be made into aqueous slurries of low viscosity and containing 15–25% by weight of pigment by use as dispersing agent of a tannic acid product, lignin or sulphonated lignin and an alkali metal or ammonium hydroxide. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *USP* 2,796,327

Methods and apparatus for producing Furnace Black, acrylonitrile and HCN in the same process, e.g. by injecting a fluid containing ammonia into the effluent smoke of a Furnace Black process. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *USP* 2,796,332

Describes ways of recovering all the pigment from the effluent gases and of using the effluent gas for the synthesis of liquid products mainly of a hydrocarbonaceous or oxygenated hydrocarbonaceous character. C.O.C.

Densifying Colour Grade Carbon Blacks
Godfrey L. Cabot *BP* 796,433

The fluffy pigment is agitated while water is sprayed on it to produce a product containing 1–12% by weight of water and a tap density of 11–16 lb. per cu. ft. It is then tumbled in a revolving, unobstructed drum until the pigment is converted into pellets of density > 16 lb. per cu. ft. The final product is remarkably free from dust and readily dispersible in ink and paint vehicles. C.O.C.

Purifying Graphite (C.I. Pigment Black 10)
Southwestern Graphite Co. *USP* 2,787,528

Graphite containing < 3% ash is obtained by treating the impure graphite with dil. H_2SO_4 and then with a mild caustic. When the graphite contains free silica a mild fluorine wash may be given but only after the caustic treatment. C.O.C.

Water-dispersible Carbon Black (C.I. Pigment Black 6 and 7)
J. M. Huber Corpn. *USP* 2,793,134

A swelling type of bentonite is mixed with the Carbon Black and the mixture densified by pressure or by rolling while either wet or dry until it forms small beads of weight > 18 lb. per cu. ft. The product disperses readily when put into water. C.O.C.

Transparent, Non-crystalline Grey Pigment
DuP *USP* 2,795,483

An amorphous grey pigment is composed of 63–71% Cu (as CuO) and 15–20% of P (as P_2O_5) and combined or adsorbed water. It may be prepared e.g. by treating aq. Na_2HPO_4 with aq. $CuSO_4$, and heating the resulting suspension of $Cu(OH)_2$ at a sufficiently high pH to ensure that black cupric oxide is formed. C.O.C.

Coloured Pigment for Camouflage Coating Compositions
National Lead Co. *USP* 2,795,511

A pigment having the spectrophotometric characteristics of chlorophyll comprises oxidic compounds of 6–65% Cr, 1–40% Co and 12–90% Ti (calculated as Cr_2O_3, CoO and TiO_2 respectively).

USP 2,796,357–8

Addition of zinc and/or antimony oxides increases the reflectance value of the above pigment and in particular the slope of the spectrophotometric curve in the critical region of the wavelength band lying between 670–750 mμ. C.O.C.

Iron Oxide Pigments
Godfrey L. Cabot *BP* 796,037

Iron is dissolved in dil. HCl, the solution evaporated to dryness and the small $FeCl_2$ particles treated with Cl gas to obtain $FeCl_3$ vapour. This vapour is hydrolysed in steam at ≮ 500°c. to yield a freely divided solid oxide. The product is of high purity and very uniform colour and particle size. C.O.C.

Finely Divided Pigments especially Berlin Blue (C.I. Pigment Blue 27)
Chemische Fabrik Wesseling *USP* 2,793,133

When the pigment is being formed precipitation is carried out in presence of 5–40% (on wt. of the final product) of a compound which forms a coating on the pigment particles and is compatible with the lacquer in which the pigment is to be used. This results in a pigment of fine particle size which is readily and firmly bound to the rubber or lacquer to which it is added. Thus 20% dibutyl phthalate (calc. on wt. of Berlin Blue to be produced) is added to a ferrocyanide solution and emulsified by vigorous stirring. A ferrous salt solution is added to this emulsion. The resulting Berlin White is oxidised by conventional methods and dried as usual. An extraordinarily finely divided Berlin Blue is produced which is excellent for use in coloured lacquers. C.O.C.

Hydrophobic, Organophilic Pigments
American Cyanamid Co. *USP* 2,789,919

Pigments are rendered suitable for use in lithographic inks by coating them with an *iso*cyanate of formula RNCO (R = aliphatic hydrocarbon of > 5 C). Thus hydrated iron oxide is slurried in dry toluene and a mixture of *n*-hexadecyl*iso*cyanate, *n*-octadecyl*iso*cyanate and *n*-octadecenyl*iso*cyanate added and the whole refluxed for 1 hr. The pigment was filtered, washed with toluene, dried and heated for 20 min. The product was satisfactorily water-repellent for lithographic needs. C.O.C.

Organo-chemical Contribution to the Inorganic Chemistry of Nitrous Acid (III p. 595)
Metal Pigments for Painting and Spraying (V p. 606)
Direct Dyeing of Cellulose. III—Dyeing Properties of Congo Red (C.I. Direct Red 28) and Concentrated Benzopurpurine 4BKX (C.I. Direct Red 2) (VIII p. 609)
Photochemistry of Aminoanthraquinones (VIII p. 610)
Measurement of Covering Power of White Pigments (XIV p. 618)

V—PAINTS; ENAMELS; INKS

Electron Microscope in the Surface-coating Industry
J. Marriott
J. Oil & Col. Chem. Assocn., **41**, 363–378 (May 1958)
The scope and the use of the electron microscope are indicated and illustrated by particular reference to studies in the structural changes in ZnS and lithopone brought about by calcination, and in the weathering of paint surfaces. Use of a technique for the preparation of dried paint films for examination by transmitted beams is described. There are 28 electron micrographs. J.W.D.

Titanium Dioxide in Polyvinyl Acetate Emulsion Paints
M. C. Londergan
Off. Dig. Fed. Paint Varn. Prod. Cl., **30**, 315–343 (March 1958)
Titanium dioxide pigments differ in apparent water demand in polyvinyl acetate (PVA) systems; this difference is more pronounced at higher pigment volumes for a given post-plasticised system. In interior paint formulations, at pigment volume concentrations (PVC) of 40, 45, and 50%, titania with higher water demand gives, weight for weight, better flat dry hiding at a given PVC. A low-water-demand rutile TiO_2 may be made to show a cost advantage over an anatase, with equality of hiding power, if some of the PVA in the system is replaced by extender. Dry flat hiding with smaller quantities of TiO_2 may be obtained with a high-water-demand anatase, for uses such as ceiling paints, where enamel hold-out, stain removal, and porosity are not critical. With exterior paints PVA films require a higher percentage of chalk-resistant rutile than do conventional oil-type films to give self-cleaning characteristics. For such paints a PVC range of 20–35% is optimal for durability, and tint retention is better with low PVC. The use of mica, combined with talc, clay, or diatomaceous silica, as extender, improves durability.
The following points arise from a subsequent panel discussion— (i) The water demand of titania depends on the surface characteristics and upon the state of agglomeration. (ii) Hexylene glycol gives the best aid to film coalescence at low temp. without increasing susceptibility of the paint itself to freezing out. Butyl carbitol acetate is effective, but it affects the freeze–thaw stability adversely. (iii) Dirt retention by the paint film is accentuated by low PVC, and by the use of too large a proportion of chalk-resistant pigment. (iv) A mica content of 0·3–0·5 lb. per gal. (U.S.A.) is recommended. (v) In calculating PVC, thickeners, surfactants, and other water-soluble materials are ignored. (vi) High-viscosity Methocel is recommended as thickener, particularly where exterior durability is in question, but high proportions of water-soluble materials are not favoured. J.W.D.

Prevention of the Thickening of Bodied Oil Enamel Lacquers pigmented with Zinc White (C.I. Pigment White 7)
M. Kühn and M. Koch
Farbe und Lack, **63**, 597–599 (1957): *Chem. Abs.*, **52**, 6810 (25 April 1958)
The thickening of ZnS bodied oil lacquers is caused by partial decomposition of the pigment to ZnO which forms salts with carboxylic groups in the polymerised unsaturated glycerides. The extent of thickening cannot be predicted from the acid number of the oil as the distribution of carboxyl between monomeric and polymeric species is decisive. Treating the oil at 100–200°c. with a condensate of 1 mol. *p*-hydroxyphenylbiguanide, 2 mol. *iso*octylphenol, 2 mol. diethylenetriamine and 6 mol. HCHO or at 250°c. with a condensate of 1 mol. phenylbiguanide and 2 mol. HCHO did not alter the acid number. However, thickening at room temperature and at 50°c. was inhibited by addition of 2% cumenesulphonamide, *NN'N''*-tris(tetrahydronaphthylsulphonyl)diethylenetriamine, tetrahydronaphthalenesulphonamide, *p*-toluenesulphonamidoacetic acid, dibutyl sulphamoylphthalate, nitromethane or mandelic acid. These additives are believed to solvate the Zn salts and so prevent their aggregation. C.O.C.

Metal Pigments for Painting and Spraying
H. Groebl
Farbe und Lack, **63**, 595–597 (1957): *Chem. Abs.*, **52**, 6808 (25 April 1958)
Al pigment (C.I. Pigment Metal 1) should have a diameter/thickness ratio of 50–150. A layer of stearic acid seems to be chemisorbed on the Al surface and provides stability to the leafing and flotation characteristics. With nonleafing-type lacquers used to obtain irridescence, the pigment is wetted by the solvent and aromatic solvents must not be used if flotation is to be avoided. The pigments should have uniform grain and the lacquer should be formulated to minimise convection. Bronze pigments must contain > 70% Cu. The binder must be of low acidity and free from unsaturated oils. Zn, Pb, and Mg pigments are also used for protecting iron from corrosion. C.O.C.

PATENTS

Phosphate Esters and their Amine Salts as Flatting Agents for Synthetic Resin Coating Compositions
DuP *USP* 2,793,196
Acid phosphate mixed esters and their amine salts, e.g. the acid phosphates of commercial primary C_{12} and C_{14} alcohols, are excellent flatting agents for use in synthetic resin coating compositions, 0·1–3·0% on the wt. of the synthetic resin giving satisfactory results. C.O.C.

Non-yellowing Stoving Finish
Armour & Co. *USP* 2,791,568
Non-yellowing alkyds suitable for plasticising melamine- and urea-formaldehyde stoving finishes consist of alkyds modified with lauric acid or a fatty acid of 8–18 C containing a substantial proportion of lauric acid or by resin acids, e.g. abietic acid stabilised against oxidation, and preferably also containing a substantial proportion of adipic acid. C.O.C.

Adsorption and Surface Characteristics of some Inorganic Pigments (IV p. 600)
Red to Maroon Azo Pigments (IV p. 602)
Hydrophobic, Organophilic Pigments (IV p. 605)
Measurement of Covering Power of White Pigments (XIV p. 618)

VI—FIBRES; YARNS; FABRICS

Influence of Weathering prior to Harvest on certain Properties of Cotton Fibres
P. B. Marsh, G. V. Merola, M. L. Butler, and M. E. Simpson
Text. Research J., **28**, 95–111 (Feb. 1958)
Most cotton bolls are subjected to weathering of from one to many weeks after they have opened and before they are collected. This weathering affects many of the fibre properties including dye absorption, content of water-soluble reducing substances, and susceptibility to enzymatic decomposition. Several of the fibre properties which change during weathering have been measured on commercial fibre samples, and the results found to show a relationship to the grade of the fibre. A rapid and practical test for measuring the water-soluble copper-reducing constituents in raw cotton fibre is described. S.B.D.

Molecular Weight of Two Seed-hair Celluloses
T. E. Timell
Text. Research J., **28**, 270–271 (March 1958)
New data are presented on the D.P. of the cellulose in milkweed floss and kapok. The new values of about 10^4 are twice and three times the previously determined values respectively, and correspond to that for the D.P. of cotton cellulose. The mol. wt. of the celluloses are $1{\cdot}7 \times 10^6$. S.B.D.

Effects of γ-Radiation on Cotton
I—Properties of Purified Cotton irradiated in Oxygen and Nitrogen Atmospheres
F. A. Blouin and J. C. Arthur
Text. Research J., **28**, 198–204 (March 1958)
Two series of samples of purified cotton were irradiated in O_2 and N_2 with γ rays at six dosage levels, and some of the chemical and physical properties of the irradiated cellulose were determined. It was found that high-energy γ irradiation of cotton resulted in carbonyl group formation, carboxyl group formation, and chain cleavage in the approx. ratios of 20 : 1 : 1; increased solubility in water and dilute alkali; decrease in the tensile strength of fibres; small but unusual changes in moisture regain; relatively unchanged infrared absorption spectra; and the same type of degradation in N_2 as in O_2, with oxygen producing only slight enhancement of the reaction.

II—Proposed Mechanism of Effects of High-energy γ-Irradiation on some of the Molecular Properties of Purified Cotton
J. C. Arthur

Ibid., 204–206

An equation $\ln P = k\ln N + K$ is derived where P is the molecular property and N the number of events or total dosage. Results tabulated in Part I are found to fit the equation when plotted on a log–log graph within the total energy range of 10^6–10^8 roentgens. S.B.D.

Molecular Weight of Flax Pectin
M. A. Sobolev and A. A. Krasivskaya

J. Appl. Chem. U.S.S.R., **31**, 129–134 (Jan. 1958)

Pyridine is the most suitable solvent for the determination of the mol. wt. of flax pectin by estimation of the specific viscosity of the nitropectin solution, prepared by nitration of the pectin with fuming HNO_3 in the presence of H_3PO_4. The mol. wt. of pectin from different layers of the stem varies greatly. For the pectin of the stem itself the mol. wt. is 128,900–221,300 acidic units, and for the fibre 15,240–33,070. The corresponding values for the D.P. are 496–790 and 54–121. T.Z.W.

Determination and Significance of Crystallite Size in Regenerated Cellulose Fibres
D. N. Tyler and N. S. Wooding

J.S.D.C., **74**, 283–291 (April 1958)

Determinations of accessibility and total crystalline content have been made for a number of regenerated cellulose fibres. Moisture sorption has been used as a measure of cellulose accessibility, and total crystalline content has been estimated from density measurements. It is suggested that the results may be interpreted as showing differences in the average cross-sectional area of crystalline regions. An attempt is made to correlate these differences with the properties of the fibres produced by various methods of manufacture. AUTHORS

Sorption of Moisture by Polymers. III—Cellulose Acetate and Cellulose Triacetate Fibres
D. K. Beever and L. Valentine

J. Textile Inst., **49**, T95–T103 (Feb. 1958)

The adsorption and (in some cases) the desorption isotherms of commercial samples of secondary cellulose acetate and cellulose triacetate from 8 firms were studied at room temperature, 22 ± 1°C. 24 samples of secondary cellulose acetate had a mean regain of 6·27% at 65% R.H.; standard deviation 0·23%, range 0·8%. 5 samples of cellulose triacetate showed a mean regain of 4·24% at 65% R.H.; standard deviation 0·24%, range 0·56%. Secondary cellulose acetate flake has a higher regain than the extrusion-spun fibre, and it is assumed that extrusion spinning causes an increase in the degree of order of the polymer and therefore a decrease in accessibility. Stretch spinning, in which Celanese (BrC) is extended 1000% to form Fortanese, causes, at R.H. > 40%, a further decrease in regain as a result of still greater ordering. However, the regain of Fortanese is sigmoidal, and the difficulty of explaining the increase in regain of Fortanese compared with Celanese at R.H. < 40% is pointed out by the authors. Denier, conversion to staple fibre, solution dyeing, and delustring have little effect on regain. P.T.S.

Microscopic Studies on Keratin Fibres
J. Menkart and A. B. Coe

Text. Research J., **28**, 218–226 (March 1958)

The distribution of cystine in the cortex of various animal fibres was investigated by staining cross-sections with mercury vapour. The receptivity to dyes of various parts of the cortex was found to be inversely related to their cystine content. After oxidation with peracetic acid or bromine the cystine-rich paracortex swells more in aqueous soln. than the orthocortex; the swelling is repressed at high salt concn. S.B.D.

Tryptophan Content of Wool
D. R. Graham and K. W. Statham

Nature, **181**, 861 (22 March 1958)

Redetermination of tryptophan in wool indicates 0·93–1·07%. Fine wools contain more tryptophan than do coarse wools. Other keratins contain 0·5–1·1%, whereas collagens contain only traces. Wool (30 mg.) is added to a solution of p-dimethylaminobenzaldehyde (100 mg.) in 18·1 N-H_2SO_4. After one week in the dark the solution is treated with 0·1 ml. of 0·01M. sodium nitrite, and the resultant blue coloration is measured photoelectrically. E.V.T.

The α–β Transformation in Keratin
A. R. B. Skertchly

Nature, **181**, 639–640 (1 March 1958)

A quantitative X-ray study of the α–β transformation in wool keratin has been made, stretching being carried out in water at 50°C. It is deduced that the reaction proceeds through an intermediate β* phase which is less ordered than either α or β phase. There is a close correlation between elastic properties and changes in X-ray diffraction intensities. The α phase is in series with a less extensible γ phase, which requires severe disulphide bond fission to enable it to be strained. The fraction of β material present at an extensibility x is given by $\beta = 1{\cdot}225x + 0{\cdot}0816$. The essential feature of the initial change is a hydration effect accompanying chain straightening. Interchain hydrogen bonding is initially absent in the β form, but appears after steam-setting and prolonged relaxation. The difference in entropy between the α and β phases is small. W.R.M.

The α–β Transformation in Keratin
H. J. Woods

Nature, **181**, 761 (15 March 1958)

The use of the index proposed by Gupta (*Nature*, **181**, 113 (11 Jan. 1958)) as a measure of the "degree of order" in the α–β transformation is criticised. It is suggested that changes in the index are indications of changes in the β/α ratio rather than in the degree of order. W.R.M.

Reactivity of Keratin
R. C. Ghosh, J. R. Holker, and J. B. Speakman

Text. Research J., **28**, 112–119 (Feb. 1958)

The amorphous phase in wool is created by the amino acids with large side-chains, amongst which are those commonly used in cross-linking reactions. Since cross-linking is unlikely to have a profound effect on the elastic properties of a disordered structure unless it occurs with great frequency, it is clear why cross-linking with agents such as formaldehyde fails to give desirable technical effects. A more promising method of modifying the properties of wool to commercial advantage is to form graft copolymers in the amorphous phase. Possible methods of achieving this end are examined. S.B.D.

Ultraviolet Irradiation and Wool Epicuticle
A. R. Haly

Text. Research J., **28**, 182–183 (Feb. 1958)

Wool fibres which had been abraded to remove all cuticular material and others which had been treated with alkali to modify the cuticle were cut in half, and one part was irradiated for a time known to give a large increase of dyeing rate to intact fibres. On examination, the dyeing rates of the unexposed and the exposed parts were approximately equal. The main effect of the irradiation of the intact wool was therefore modification of the cuticle. This did not destroy the epicuticle, as immersion in saturated bromine water still gave Allwörden blisters, although their time of formation was much increased. It is suggested that the tyrosine residues are affected and that the felting properties may be modified. S.B.D.

Glass Transition of 6- and 6,6-Nylons
F. Rybnikar

J. Polymer Sci., **28**, 633–634 (April 1958)

Glass transition temperatures, measured by a penetrometer method, are −65° and −66°C. for two samples of 6-nylon, and −65·5°C. for a sample of 6,6-nylon. These are much lower than values previously reported, which are probably related to changes in crystalline structure or to temperatures of viscous flow of the amorphous phase. W.R.M.

Photochemical Degradation of 6-Nylon—I
T. Hashimoto

Bull. Chem. Soc. Japan, **30**, 950–952 (Dec. 1957)

Following exposure of 6-nylon fabric in a Fade-Ometer, determination of the water-soluble material and the intrinsic viscosity of the residue shows that the chains are broken randomly. The measurements are consistent with current theories of degradation by random scission of the polymer chains. E.V.T.

Changes of the Intermolecular Structure of Capron (6-Nylon) on Heating
L. I. Pokrovskii and A. B. Pakshver
Colloid J. U.S.S.R., **19**, 478–482 (July–Aug. 1957)
The apparent heats of dissolution in concentrated formic acid of (*a*) unstretched, (*b*) stretched Capron fibre which had been maintained at 140 and 160°C. respectively for various lengths of time are measured and lower values reported in case (*b*). This difference is attributed to changes in the molecular packing and density of the fibre. G.J.K.

Occlusion of High Polymers. II— Changing the Properties of Polyamide Fibres by the Occlusion Process
A. N. Bykov, M. I. Ivanova, and A. B. Pakshver
Colloid J. U.S.S.R., **19**, 542–547 (Sept.–Oct. 1957)
The occlusion of benzene within stretched and unstretched Capron (6-nylon) is studied. It is found that occlusion accelerates many times the diffusion of reagents into the bulk of the fibre and is of special importance for increasing the rate of a process, such as drying, which is determined by internal diffusion. The more rapidly the swelling agent (20 g. phenol per litre) in the polymer is replaced by benzene, the greater the reactivity of the polyamide fibre. For given conditions, the increase in polyamide reactivity attained in the occlusion process is independent of the degree of drying of the occluded substance, of time, and of the temp. at which occlusion occurs. G.J.K.

PATENTS

Rendering Viscose Rayon Flame-resistant
R. S. Robe *BP* 795,133
Viscose rayon containing a minor proportion of cellulose having a reactive carboxymethyl, glycerol ether or carboxyethyl group is treated after extrusion and before it is dried with a compound of a metal which has flame-resisting properties and which forms an insoluble compound with the reactive group. Thus viscose containing 0·8% of high viscosity carboxymethyl cellulose was formed into filaments in the usual manner, acidified to ensure presence of free acid carboxy groups, and then immersed in 6% aq. potassium dihydro pyroantimonate for 30 min. at 55°C., immediately transferred into 20% aq. ammonium zirconyl carbonate for 30 min. at 55°C. rinsed and dried. It had excellent flame resistance even after leaching and repeated laundering. C.O.C.

Imparting Affinity for Acid Dyes to Cellulose Acetate Fibres
Celanese Corpn. of America *USP* 2,789,916
Incorporation of an epoxyamine or its hydrolysis product, an aminediol or an alkanolamine containing a tertiary N atom, yields a product which dyes readily with acid dyes to give dyeings having good fastness to washing. These agents may be added to the spinning dope or applied to acetate rayon from solution in a suitable solvent. C.O.C.

Solutions of Dextran in Aqueous Boron Trifluoride as Textile Finishes and for Producing Fibres (III p. 596)
Reactivity of Cellulose (XI p. 615)
Copolymerisation of ε-Caprolactam with 2-Vinylpyridine or 2-Methyl-6-vinylpyridine in Bulk and in Solution (XIII p. 617)
Determination of Apparent Second-order Transition Temperatures of Polymers (XIV p. 620)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Foam Stability
A. J. de Vries

I— Structure and Foam Stability
Rec. Trav. chim., **77**, 81–91 (Feb. 1958)
A critical, thermodynamic survey of the factors governing foam stability.

II— Gas Diffusion in Foams
Ibid., 209–223 (March 1958)

III— Spontaneous Foam Destabilisation resulting from Gas Diffusion
Ibid., 283–296 (April 1958)

IV— Kinetics and Activation Energy of Film Rupture
Ibid., 383–399 (May 1958)
E.V.T.

Relation between Viscosity and Critical Micelle Concentration of Detergent Solutions. III— Relations between Micellar Molecular Weight, Viscosity, and Critical Micelle Concentration of Detergent Solutions
K. Tyuzyo
Bull. Chem. Soc. Japan, **31**, 117–122 (Jan. 1958)
Association number n and critical micelle concn. C_c for a number of anionic and cationic detergents are related by the expression—

$$n = 17{\cdot}0\left(\frac{1}{C_c}\right)^{0{\cdot}29}$$

independently of added electrolytes. Micellar molecular weight M_m and intrinsic viscosity $[\eta_1]$ and $[\eta_2]$ of both ionic and non-ionic detergents are related by the expressions—

$$\log M_m = 0{\cdot}41\{\log n[\eta_1]\}^2 - 0{\cdot}39\{\log n[\eta_1]\} + 3{\cdot}47$$
$$= 0{\cdot}45\{\log n[\eta_2]\}^2 - 0{\cdot}91\{\log n[\eta_2]\} + 4{\cdot}04$$

For definitions and explanations of $[\eta_1]$ and $[\eta_2]$ see Sata and Tyuzyu, *ibid.*, **26**, 177 (1953). A.J.

Neutral Scouring of Greasy Wool
E. C. Hansen
Amer. Dyestuff Rep., **47**, P 155–P 156 and P 159 (10 March 1958)
A nonylphenol-base detergent containing 10·5 moles of ethylene oxide is used as a non-ionic detergent for scouring raw wool. The product is whiter and loftier than wool scoured conventionally. The method can be used with any kind of wool or mohair or camel-hair, and the quantities required for three-, four-, or five-bowl scouring are given. The wool grease produced by this process is darker, and further research in scouring is not ignoring the quality of wool grease produced or the possibilities of solvent scouring for wool. P.T.S.

Laundering Procedures in the Commercial Laundry and in the Home
AATCC Committee on Dimensional Changes in Textile Fabrics
Amer. Dyestuff Rep., **47**, P 187–P 190 (24 March 1958)
This article analyses the methods used in commercial laundries to wash heavily soiled white fabrics excluding wool, to wash fast colours, to wash moderately fast colours, and to wash wool. Home laundering is discussed, and the aftertreatments (drying, ironing, pressing) at home and at the laundry are mentioned. The information was assembled as a guide to future work on the dimensional stability of cloth during laundering. P.T.S.

Condensed Phosphates in Textile Processing
K. Schuster
Textil Praxis, **13**, 387–393 (April 1958)
A survey is presented of the application of condensed phosphates in textile finishing with special reference to Calgon. Its use as a metal-sequestering, dispersing, peptising, and buffering agent is described in some detail. L.A.T.

Application of Calgon in Bleaching
K. Schuster and J. Umlauf
Textil Praxis, **13**, 396–399 (April 1958)
Improved whiteness, reduced possibility of catalytic damage, and other benefits are derived from the use of Calgon T in the bleaching of textiles with sodium hypochlorite, sodium chlorite, and hydrogen peroxide. L.A.T.

Effect of Iron and Copper Contaminants on Cotton Degradation in Peroxide Bleaching
AATCC Southeastern Section
Amer. Dyestuff Rep., **47**, P 79–P 83 (10 Feb. 1958)
In a preliminary study of the problem, tests were made with fabrics impregnated with contaminants before being bleached and with bleach-baths containing contaminants. The effect of degradation was measured by skein breakage tests and cupriethylenediamine viscosity measurements. The investigation is not complete, but contamination of bleach baths by metal oxides or salts does not necessarily damage cotton; for example, ferric oxide as a contaminant does not weaken cotton more than uncontaminated peroxide, and thus rust does not seem to be a source of danger. Solid particles of contaminant may cause local

overheating by catalysis of peroxide decomposition. 18 references are given, some to the catalysed decomposition of hydrogen peroxide. P.T.S.

Bleaching of Groundwood Pulp with Combinations of Peroxide and Hydrosulphite
R. W. Barton
T.A.P.P.I., **41**, 161A–165A (March 1958)
The brightness increases possible with either of the two agents individually are limited. With peroxides, the max. practical gains are about 14 points and with hydrosulphites about 12 points. To increase the brightness 1 point above these limits with either process alone would entail the expenditure of prohibitive amounts of money in chemicals. By using a combination of the two agents, the brightness gains are within limits additive. Experimental results for spruce groundwood are given, but the process may be applied to any type of pulp. R.A.

PATENT

Removing Burn or Scorch Marks from Fabric
M. Broos *USP* 2,792,277
The scorched area is covered with a powdered mixture of 92% paraffin wax and 8% zinc oxide, moistened with 10 vol. H_2O_2, rolled up and stood for 1 hr. If necessary the treatment is repeated. C.O.C.

Influence of Dyes on the Degradation of Cotton by Washing and Bleaching (VIII below)
Application of Yarn Hairiness Measurement Techniques to the Control of Gassing or Singeing (XIV p. 619)

VIII—DYEING

Critical Appraisal of Dyeing Processes
J. Boulton
Canadian Textile J., **75**, 57–59 (21 March 1958)

Permeability and the Size Distribution of Pores
M. R. J. Wyllie and G. H. F. Gardner; T. J. Marshall
Nature, **181**, 477 (15 Feb. 1958)

Dyeing and Finishing of Warp-knitted Fabrics of Low Elasticity
W. Ernst
Textil Praxis, **13**, 399–401 (April 1958)
Procedures are described for handling warp knitted fabrics in tubular form. Emphasis is laid on the prevention of creasing or excessive stretching of the fabric. L.A.T.

Dyeing Properties of Mature and Immature Cotton
N.-B. Furvik
J.S.D.C., **74**, 299–303 (April 1958)
Laboratory dyeing experiments, carried out with direct dyes on immature and mature cotton fibres of known origin, showed that the maturity did not affect the amount of dye absorbed at equilibrium. The absorption and desorption rates, however, were higher for immature than for mature fibres. Mercerisation of the fibres before dyeing did not greatly affect the differences. The causes of why immature fibres in general and especially in neps look lighter in colour than the rest of the fabric are discussed. A theory is put forward that, besides the effect of optical differences, the differences in depth are caused by the more rapid desorption of dye from immature fibres. Such differences would be exaggerated by unsuitable processing conditions, e.g. during rinsing, which was confirmed by some laboratory dyeings on fabric samples. From the results, it was predicted that, in order to obtain an even colour on a fabric containing immature as well as mature fibres, dyeing should not be carried on until equilibrium is reached and that rinsing and aftertreatments should be carried out so that the dye desorption will be as small as possible. Author

Influence of Dyes on the Degradation of Cotton by Washing and Bleaching
J. Cyrot and J. Bouligaud
Bull. Inst. Text. France (70), 7–19 (Oct. 1957)
Exposure in the Fade-Ometer of samples of cotton fabric dyed with direct and vat dyes, followed by determination of the D.P. of the cellulose, confirmed earlier findings that certain yellow and orange dyes increase the rate of degradation of cellulose by light, whilst some blues and greens exert a slight protective effect. Other samples of the same dyed fabrics were given repeated launderings (washing, hypochlorite bleaching, and ironing). An increased rate of degradation was found for the samples dyed with yellow direct dyes. The other dyes examined were without effect with the exception of Solanthrene Brilliant Green 2F (C.I. 59825), which appeared to exert a slight protective effect. J.C.F.

Effect of Nuclear Radiation on Fibrous Materials. II—Dyeing Characteristics of Irradiated Cotton and Rayon
O. Teszler, H. Wiehart, and H. A. Rutherford
Text. Research J., **28**, 131–135 (Feb. 1958)
The dye-absorption characteristics of a series of cotton and rayon samples exposed to γ-rays and thermal neutrons for different periods of time have been determined, using dyes of known behaviour towards degraded celluloses. At the early stages and again during the later stages of neutron irradiation, the dye absorption was increased; at intermediate exposures the absorption was decreased. Because hydrolysed cellulose behaves in the former manner and the acidic type of oxidised cellulose in the latter, the data suggest that neutrons have a similar effect to both hydrolysis and oxidation. γ-Rays from a ^{60}Co source gave only the effect of oxidation. S.B.D.

State of Direct Dyes in Solution and their Diffusion into a Cellophane Membrane
B. N. Mel'nikov and P. V. Morӯganov
Colloid J. U.S.S.R., **19**, 689–695 (Nov.–Dec. 1957)
A definite correlation exists between the state of aggregation of direct dyes in soln. and their rate of diffusion into a Cellophane membrane, the rate of diffusion increasing roughly tenfold in the presence of a dispersing agent. This effect is strongest at low temp. and at the optimum concn. of agent in the soln. Diazo Black C (C.I. 401), Sky Blue K (C.I. 406), Diazo Black O (C.I. 395), and Sky Blue (C.I. 518) were investigated using pyridine as the dispersing agent. The diffusion coefficients were determined by the method of Northop and Anson (*J. Gen. Physiol.*, **12**, 543 (1929)). G.J.K.

Direct Dyeing of Cellulose. III—Dyeing Properties of Congo Red (C.I. Direct Red 28) and Concentrated Benzopurpurine 4BKX (C.I. Direct Red 2)
K. Nishida and I. Sugiyama
Sen-i Gakkaisha, **11**, 744–749 (1955):
Chem. Abs., **52**, 6798 (25 April 1958)
The absorption isotherms of C.I. Direct Red 28 and 2 obtained at 90°c. electrophotometrically from baths containing 0·05–0·10 × 10^{-3} dye and 0·05–3·0 mole NaCl per kg., showed that presence of CH_3 groups in the dye molecule increased and that of SO_3Na groups decreased the affinity for cotton. The aggregating tendency of the dyes increased with their affinity for cellulose. The dyeing properties decreased markedly by the effect of salting-out by the electrolyte. C.O.C.

Mode of Action of Non-ionic Levelling Agents
W. Luck
J.S.D.C., **74**, 221–235 (April 1958)
Successful results in dyeing depend on a series of simultaneous equilibria. The formation of micelles by non-ionic levelling agents has been demonstrated spectroscopically and the micellar size determined. The equilibrium conditions in the formation of complexes between dyes and levelling-agent micelles have been studied. These conditions are intimately related to the equilibrium between dye and fibre. The latter equilibrium has been investigated with dyeings on polyamides. Study of the simultaneous equilibria dye–fibre and dye–levelling agent gives a clear picture of the nature of the retarding and levelling action of textile auxiliaries. The equilibrium between auxiliary and dye decreases the association of the latter. Results obtained in previous diffusion studies, however, have been interpreted as indicating increased association of dyes on addition of levelling agents. These results have been re-examined, and shown to be in accordance with the theories developed in the present paper. Author

Reactive Dyes on Cellulosic Fibres
T. Vickerstaff
Amer. Dyestuff Rep., **47**, 33–38 (27 Jan. 1958)
This paper gives the theory of dyeing with Procion (ICI) reactive dyes. These are simple monoazo anthraquinone or phthalocyanine derivatives which are polysulphonated to give high water solubility. With the exception of the phthalocyanine derivative they are of small molecular size and diffuse rapidly within the fibre. They are therefore

suitable for cold dyeing and continuous processing. The dyes owe their wet fastness to the fact that in alkaline solution they can combine chemically with hydroxyl groups of cellulose to give a stable bond. There are two competing reactions—

$$NaSO_3\text{-D-Cl} + HO\text{-Cellulose} \rightarrow NaSO_3\text{-D-O-Cellulose} + HCl$$

$$NaSO_3\text{-D-Cl} + H_2O \rightarrow NaSO_3\text{-D-OH} + HCl$$

The second reaction is reduced in importance by applying the dye in neutral solution and then making the conditions alkaline, when the reaction of the dye with cellulose takes place immeasurably fast, and loss of dye by reaction with water is minimised. The theory of dyeing with reactive dyes is considered in terms of the probability of a reactive dye molecule diffusing into the fibre before it can react with a hydroxyl ion. P.T.S.

Application of Reactive Dyes to Viscose Rayon
J. A. Fowler and C. Preston
J.S.D.C., **74**, 372–381 (May 1958)

The rate of fixation of Procion (ICI) dyes can be made to vary from slow at room temperature and mild alkalinity to rapid at high temperature or high alkalinity, intermediate rates being achievable at will by controlling various simple factors. Choice of method of dyeing is therefore wide and will depend largely on the particular equipment readily available. AUTHORS

Adsorption of a Reactive Dye by Modified Celluloses
E. H. Daruwalla and P. Subramaniam
J.S.D.C., **74**, 296–299 (April 1958)

The equilibrium adsorption of commercial Procion Brilliant Red 2BS by acid-modified cellulosic fibres and by standard cellulose altered by the specific action of different oxidising agents is studied. The equilibrium adsorption by all the fibres decreases rapidly in the initial stages of the action of acid, but tends to level off with further hydrolysis. The extent of the decrease depends mainly on the rate of removal of the hydrolysable fraction and the extent of crystallisation of the cellulose chains during the action of the acid. The reduction in dye uptake is more marked when primary alcoholic groups are converted into carboxyls than when secondary alcoholic groups are similarly oxidised. AUTHORS

New Developments in Dyeing with Cibacron (Ciba) Reactive Dyes
M. Peter
S.V.F. Fachorgan, **13**, 184–193 (April 1958)

In the absence of electrolytes the dyes exhibit virtually no substantivity for cellulosic fibres, and may thus be considered very suitable for padding. The following methods of piece dyeing are described: pad–jig, Pad–Roll, pad–steam, single pad–steam and pad–dry heat fixation. The dye not chemically bound to the fibre is removed by a thorough rinse and hot soaping. Cibacron dyes can also be applied to yarns and fabrics by exhaustion in the presence of electrolyte. Dye fixation is affected by the addition of alkali. Cibacron dyes possess good all-round fastness properties. L.A.T.

X-Ray Study of the Compounds formed by Basic Dyes with Acidic Oxycelluloses and Alginic Acid
J. O. Warwicker
J. Textile Inst., **49**, T 148–T 169 (March 1958)

The X-ray diffraction pattern from oxycellulose dyed with Methylene Blue (C.I. Basic Blue 9) shows the pattern of undyed oxycellulose and, in addition, a line which is identical with a strong line which appears in the diffraction pattern from Methylene Blue chloride. When cellulose is oxidised to oxycellulose, carboxyl groups are formed. Salt formation can occur between these groups and the Methylene Blue cations. The original suggestion (T. P. Nevell, *ibid.*, **42**, T 130 (1951)) to explain the appearance of the line from the dye diffraction pattern in the pattern from the dyed fibre was that Methylene Blue cations clustered around multivalent micellar anions in a crystalline or semi-crystalline way. The present extension of the work indicates that the new line in the diffraction pattern from the dyed fibre is caused by pairs of dye molecules attached to different cellulose chains which have achieved, by the operation of electrostatic and dipolar forces, the same separation as in the dye crystal. P.T.S.

Dyeing Hydrophobic Fibres in Solutions of Solvents
M. K. Gokhale, L. Peters, and C. B. Stevens
J.S.D.C., **74**, 236–241 (April 1958)

Data are presented concerning the rate of dyeing of cellulose triacetate loose staple fibre with 3% Duranol Red X3B 300 (C.I. Disperse Red 11, C.I. 62015) from aqueous solutions of *n*-butanol or benzyl alcohol. In water saturated with *n*-butanol, more than 85% exhaustion is reached after 1 hr. at 80°C., 30 min. at 60°C., or 1 hr. at 40°C. After 30 min. in water saturated with benzyl alcohol, 68% of the dye present is fixed on the fibre at 40°C., 78% at 60°C., and 80% at 80°C. In general, with either solvent, the highest amount of dye fixation is obtained at about 60°C. At higher temperatures less solvent is needed and the time of dyeing can be reduced. A method for dyeing Tricel (BrC) or Terylene (ICI) fabric is suggested. AUTHORS

Recent Developments in Carpet Dyeing
W. J. Wygand
Amer. Dyestuff Rep., **46**, P 987–P 990 (30 Dec. 1957)

Nylon, rayon, cotton, and, lately, acrylic fibres are being used as well as wool for carpet yarns. Neutral-dyeing metal-complex dyes for wool have excellent dyeing and fastness properties. They can be applied by a continuous process to wool and nylon. Direct dyes have been used for rayon; for rayon–wool blends selected direct and neutral-dyeing metal-complex are used. Vat dyes cannot be applied economically to rayon in an open scouring bowl because the large surface area leads to excessive consumption of hydrosulphite. This has been overcome in the closed du Pont Flowstock Process. Practical details of the procedures are given. The pad–steam dyeing process will probably soon be commercially viable. P.T.S.

Photochemistry of Aminoanthraquinones
G. S. Egerton and A. G. Roach

I— Absorption Spectra in Solution and in the Solid State
J.S.D.C., **74**, 401–407 (May 1958)

The ultraviolet and visible absorption spectra of six aminoanthraquinones have been determined in solution, on polymer films, and as solid deposits on quartz. The compounds were 1- and 2-amino-, 1:4- and 1:5-diamino-, 1:4:5-triamino-, and 1:4:5:8-tetra-amino-anthraquinone. The solvents were ethyl and *iso*propyl alcohols, and transparent polymer films of nylon, *N*-methoxymethylnylon, and secondary cellulose acetate were used. The effect of the solvent or substrate on the absorption spectrum and the problem of colour and chemical constitution in relation to the aminoanthraquinones have been discussed.

II— Photochemistry of Dyed Polymer Films
Ibid., 408–414

A study has been made of the action of near ultraviolet radiation and visible light on the transparent polymer films dyed with the six aminoanthraquinones. The reaction was followed spectrophotometrically in a closed system of dry nitrogen or oxygen. The general feature of the exposures in oxygen is a decrease in optical density throughout the spectrum. The rate of fading of 2-aminoanthraquinone on nylon and cellulose acetate film is as great in nitrogen as in oxygen, although with 1:5-diaminoanthraquinone the rate is greater in oxygen. The light fastness of the aminoanthraquinones is greatest on cellulose acetate film, intermediate on nylon, and least on *N*-methoxymethylnylon. Among the six aminoanthraquinones the light fastness on a particular substrate is least with 2-amino- and 1:5-diamino-anthraquinone, and greatest with the 1, 1:4, 1:4:5, and 1:4:5:8 compounds. On exposure in nitrogen, but not in oxygen, of 1-amino- and 1:5-diamino-anthraquinone on *N*-methoxymethylnylon, a new peak around 400 mμ. is formed. Under the same conditions 2-aminoanthraquinone behaves differently.

III— Photochemistry in the Solid State
Ibid., 415–420

Solid transparent films on quartz of the six aminoanthraquinones have been prepared by vacuum deposition. The action of (*a*) near ultraviolet radiation and visible light and (*b*) short-wave ultraviolet radiation (mainly 253·7 mμ.) on these solid films has been studied spectrophotometrically. The stability of the solid dye to (*a*) is shown to be substantially greater than when the dye is present in the form

of a dyeing on a polymer film. In moist air the photochemical stability of the solid dye is much less than in dry oxygen or dry nitrogen. The nature of the changes occurring as a result of irradiation in these atmospheres is also different, and reasons for these differences are discussed. The action of (b) on the solid dye is shown to lead to rapid destruction of the dye. The rate is greater in dry oxygen than in dry nitrogen, and ultimately all the dye and photoproducts appear to be removed from the quartz. 2-Aminoanthraquinone is most rapidly affected by (a) but is the most stable of the six compounds to (b). AUTHORS

Esters of Leuco Vat Dyes. III—Oxidation of the Mono- and Di-sulphuric Esters of Quinol (Hydroquinone) and of 1:4-Naphthaquinol with Acidic Hydrogen Peroxide
A. Johnson and M. L. Rahman
J.S.D.C., **74**, 291–296 (April 1958)
The mono- and di-sulphuric esters of 1:4-naphthaquinol react with acidic hydrogen peroxide in the same way as the disulphuric ester of 9:10-anthraquinol-2-sulphonic acid—i.e. removal of most of the sulphate groups by direct oxidation follows removal of some groups by hydrolysis. The catalytic effect of the mono-ester in the oxidation of the di-ester has been directly demonstrated. On the other hand, with the mono- and di-sulphuric esters of quinol, the sulphate groups are not removed by direct oxidation but by hydrolysis only. Oxidation of the resultant quinol then follows as a separate reaction. AUTHORS

Crystallisation of Indanthrone
A. Lorincz
Magyar Kem. Folyóirat, **60**, 129–136 (1954):
Chem. Abs., **52**, 6797 (25 April 1958)
The di-Na leuco compounds of indanthrone dyes tend to crystallise in the dye bath resulting in loss of dye and lighter and duller dyeings. Unless an anticrystallisation agent is added to the vat the prescribed conditions for dyeing must be carefully adhered to. C.O.C.

Equilibria on Nylon of Anionic Dyes based on α-Naphthylamine→β-Naphthol
H. J. Palmer
J. Textile Inst., **49**, T 33–T 39 (Jan. 1958)
Data are given for equilibrium uptake of sulphonated α-naphthylamine→β-naphthol dyes by 6,6-nylon, variables being degree of sulphonation (1–5) of the dye molecule; primary amine, *N*-alkylmorpholine, and benziminazole end-group content of the nylon; pH of the dyebath. Undrawn 10-denier/filament yarn was used. Dye uptake falls with increasing degree of sulphonation, increasing pH of the dyebath, and decreasing basicity of the end-group. Dye uptake is not stoichiometric. Substitution in one 8-position of the naphthalene residues restricts rotation about the azo group; when both 8-positions are substituted, the molecule becomes non-planar. Uptake of trisulphonated dyes is lowest if both 8-positions have been sulphonated, greatest if neither has been sulphonated, and intermediate if only one has. P.T.S.

Dyeing of Filament Nylon with Acid Dyes
J. A. Bittles, J. A. Brooks, J. J. Iannarone, and H. P. Landerl
Amer. Dyestuff Rep.,
47, P 183–P 186 and P 194 (24 March 1958)
Disperse dyes, which have excellent levelling properties on nylon, cannot meet the demand for medium and dark colours because they lack the required wash fastness. Anionic dyes are fast to washing, but they have poor levelling properties, and this leads to streakiness. This can be overcome in various ways. Solvent-assisted dyeing is not economically feasible, and it is hazardous. With certain dyes, a cationic agent added to the dyebath can give excellent levelness below the boil. Anionic agents are not so satisfactory below the boil. Above boiling point, anionic and cationic agents produce level results, the anionic agent being slightly better. Procedures and mechanisms are discussed. P.T.S.

Dyeing Nylon with Mixtures of Acid Dyes
E. Atherton, D. A. Downey, and R. H. Peters
J.S.D.C., **74**, 242–251 (April 1958)
Recently published work on the uptake of acid dyes by nylon is discussed. The concept of anion activity gradient as the driving force for diffusion of dyes in nylon is applied to the uptake of mixtures of acid dyes and the problem of compatibility. It is shown that the properties of single acid dyes in finite dyebaths may be represented by "compatibility indices" and that incompatibility is entirely determined by liquor ratio. AUTHORS

Dyeing of Nylon with Acid and Mordant Dyes at High Temperatures
H. R. Hadfield and H. Seaman
J.S.D.C., **74**, 392–400 (May 1958)
A study has been made of the stability of acid and chrome dyes in solution at temperatures above 100°C. The effects of an increase in dyeing temperature above 100°C. on the levelling of acid dyes and upon the rate of chroming of chrome dyes have also been determined. The results emphasise the importance of controlling the exhaustion of the dyebath during the dyeing of nylon and confirm the rapid rate of chroming above 100°C. AUTHORS

High-temperature Dyeing of Continuous-filament Yarns of Man-made Fibres
J. Fowler and K. Walsh
J.S.D.C., **74**, 390–392 (May 1958)
The theoretical advantages of high-temperature dyeing are now generally recognised. Some of the practical aspects and the problems encountered are discussed on the basis of several years' experience in this specialised field. AUTHORS

Dyeing of Continuous-filament Nylon with Disperse and Anionic Dyes
H. W. Peters and J. C. Turner
J.S.D.C., **74**, 252–282 (April 1958)
The nature of the variations in continuous-filament nylon and their influence on dyeing behaviour are explained. By the use of a special fabric containing yarn differing in physical and chemical properties by known and controlled amounts, the sensitivities to such variations of many dyes for nylon have been assessed. These results appear in the appendix in tabular form, and are intended to help the dyer to select suitable dyes, by indicating their abilities to cover yarn irregularities. New and current dyeing methods are discussed, and suggestions are made for improved uniformity. Laboratory investigations have shown which new methods offer most prospects of success, and also where more detailed investigation will be of value. AUTHORS

High-temperature Disperse Dyeing of Terylene Polyester Fibre
H. R. Hadfield and R. Broadhurst
J.S.D.C., **74**, 387–390 (May 1958)
The building-up and the levelling properties on Terylene of a fast and a slowly diffusing disperse dye have been compared over the temperature range 85–140°C. Both properties improve rapidly as the temperature of the dyebath is raised above 100°C. Optimum dyeing conditions are attained between 120° and 130°C., and optimum levelling conditions between 130° and 140°C. Under high-temperature dyeing conditions the slowly diffusing dye gives the better dyebath exhaustion and the rapidly diffusing dye has the better levelling properties. AUTHORS

High-temperature Dyeing of Terylene Slubbing or Loose Stock
Fred Smith
J.S.D.C., **74**, 382–387 (May 1958)
Practical aspects of the operation of machines designed for dyeing loose fibres at temperatures above the boil are discussed, special stress being laid on safety precautions. AUTHOR

Colouring of Vinylon
S. Nomura and K. Tanabe
J.S.D.C., **74**, 359–371 (May 1958)
Vinylon is usually made by spinning an aqueous solution of polyvinyl alcohol, heat-treating the water-soluble fibres obtained, and subsequently acetalising with formaldehyde. As both hydroxyl and acetal groups in the accessible region are concerned in the dyeing of vinylon, it is generally intermediate in dyeing behaviour between cellulosic fibres and typical synthetic fibres. The dyeing properties of vinylon with direct dyes are thus affected not only by

crystallisation due to heat-treatment and degree of acetalisation but also by the acetalising conditions. For the practical dyeing of vinylon to medium or heavy depths direct, suphur, vat, azoic, disperse, and metal-complex dyes may be employed, and their fastness is not greatly different from their corresponding fastness on other fibres. Dope dyeing is now most advantageous for blacks and dark blues, and is carried out on a commercial scale. AUTHORS

Dyeing of Zefran and Blends containing Zefran
A. S. Messer

Amer. Dyestuff Rep., **47**, P 84–P 92 (10 Feb. 1958)

Zefran is the Dow Chemical Co.'s first synthetic fibre. It is a "nitrile alloy fibre", presumably an acrylic copolymer. Its great advantage is its ability to be dyed in blends with wool and cellulosic fibres under the usual conditions and with the same dyes as are used with these fibres. Like a cellulosic fibre it can be dyed with vat dyes, soluble vat dyes, azoic dyes, sulphur dyes, and after-treated direct dyes. For blends with wool, neutral dyeing metal-complex dyes can be used, with a levelling agent. The paper gives details of experiments on dye migration and levelling, and details of the practical application of the above dyes to blends. P.T.S.

Production of Multicolour Effects on Anodised Aluminium
V. F. Henley

Trans. Inst. Met. Finishing, **35**, 91–100 (1958)

A review of methods for producing multicolour and single-colour patterns on anodised aluminium. A.J.

PATENTS

Increasing the Affinity of Acrylic Fibres for Acid Dyes
Pacific Mills *USP* 2,792,276

The affinity of acrylic fibres for acid dyes is increased by heating them with aqueous hydroxylamine at 120°F. at pH $\nless$ 7·0. C.O.C.

Dyeing Acrylic Fibres using a Cupric Salt-Phenyl Phenol Reaction Product
Deering Milliken Research Corpn. *USP* 2,796,318

The fibre is dyed in an aqueous bath containing an acid dye, a cupric salt and a phenyl phenol which has been given a preliminary boil. This preliminary boil results in formation of a complex between the cupric salt and the phenyl phenol. Probably this complex attaches itself to the acrylic fibre and so causes affinity for the dye. There is little or no reduction of cupric ions to cuprous ions. Thus a dyebath was made up of 600 ml. water, Roracyl Dark Green B (DuP) (6 g.), Anthraquinone Blue SWF (C.I. Acid Blue 25) (1·5), Du Pont Orange RO (C.I. Acid Orange 8) (0·375), $CuSO_4$ (5·5) and *p*-phenyl phenol (3). This bath was boiled under a pressure of 15 lb. per sq.in. for 20 min. Acrylic fibre fabric was then immersed in the boiling bath under atmospheric pressure and was dyed an excellent black in 15 min. When dyed for 1 hr. excellent penetration was obtained and the dyeing was exceptionally fast to light and washing. C.O.C.

Phosphorescent Yarn
Firth Carpet Co. *USP* 2,787,558

Yarn of any fibre is impregnated in a bath containing dye, a phosphorescent pigment, a binder and a resinous substance. Thus carpet yarn is impregnated with a dispersion made up of ZnS with phosphorescent base (30 parts by wt.), casein solution (10), poly(vinyl acetate) emulsion (5), and water (55) and dried at 270°F. The casein solution consists of casein (100), water (700), borax (14·5) and Na pentachlorophenate (2·5). C.O.C.

Influence of Weathering prior to Harvest on certain Properties of Cotton Fibres (VI p. 606)

Microscopic Studies on Keratin Fibres (VI p. 607)

Relation of the Effect of Resins on Light Fading and the Tendering Action of Photosensitive Vat Dyes (X p. 613)

Improving the Fastness to Washing of Disperse Dyes on Acetate Rayon (X p. 615)

IX—PRINTING

Migration of Dyes in the Fibre-substance during Steaming
H. B. Hallows and H. A. Turner

J.S.D.C., **74**, 345–358 (May 1958)

An account is given of the examination in cross-section of sheets of fibre-substance—cellulose and secondary cellulose acetate—which have been steamed for various times in contact with a layer of thickening agent containing a dye. A variety of dyes have been used, and, from the examination, some information about the migration of dye in both thickening agent and fibre-substance film has been obtained. With some of the cross-sections an attempt has been made to measure quantitatively the distribution of dye within the fibre-substance at various times of steaming, and hence to deduce the diffusion characteristics. AUTHORS

Flash-ageing of Vat-printed Viscose Rayon Fabrics
A. S. Fern and W. F. Liquorice

J.S.D.C., **74**, 331–344 (May 1958)

The evolution of pad–steam processes for fixing printed vat dyes on cellulosic fabrics is traced. The behaviour of a wide range of vat dyes under laboratory "flash-ageing" conditions with sodium dithionite(hydrosulphite)-caustic soda as the reducing system indicated the most satisfactory thickening agent, dyes, and padding and steaming conditions, and led to the design of a bulk-scale steamer operating at about 7 yd./min. with a steaming time of about 20 sec. The percentage fixation of a selected range of dyes on viscose rayon and on cotton under flash-ageing conditions compares favourably with that obtained by the "all-in" sulphoxylate–carbonate printing process, where the steaming time is sixty times as long. Many vat dyes show improved brightness and/or colour value when steamed for short times, owing to the absence of leuco decomposition or over-reduction. A modified process is being worked out with thiourea dioxide as reducing agent in the printing paste, development being by padding with caustic soda before steaming. Preliminary experiments indicate that emulsion thickenings are unlikely to show much technological advantage over conventional thickenings in the flash-ageing process. AUTHORS

New Developments in Printing with Cibacron (Ciba) Reactive Dyes
H. Werdenberg

S.V.F. Fachorgan, **13**, 193–199 (April 1958)

The application of Cibacron dyes in direct, discharge, and resist printing is described. Reference is made to the preparation and stability of printing pastes. The printing of Cibacron dyes alongside dyes of other classes is discussed. L.A.T.

Printing with Emulsions
R. D. Greene

Amer. Dyestuff Rep., **47**, P 191–P 193 (24 March 1958)

Emulsions are used to increase the viscosity in resin-bonded pigment printing. Ideally, a single emulsion would be suitable for all classes of dye, and this is an object of printing research. Meanwhile different emulsions are used for vat, azoic, direct, and aniline black dyes. Oil-in-water emulsions are of more practical value in printing than water-in-oil. The conditions encountered using each type of dye are described, the emulsion to be used being dictated by these conditions. P.T.S.

PATENTS

Diazo Print Process
General Aniline *USP* 2,789,904

A base coated with a light-sensitive diazo compound is exposed to light and then pressed against a fibrous material impregnated with a coupler which on the assembly being heated in presence of vapour transfers to the other component where it reacts with the diazo compound. Thus paper coated with 4-benzoylamido-2:5-diethoxybenzene diazonium chloride zinc chloride double salt is exposed to light and then heated for 36 sec. at 165°C. in direct contact with a sheet of newsprint impregnated with a 20% solution of phloroglucinol in glycerol. This yielded a deep black image of good fastness to light and a pure white ground. C.O.C.

Colour Photography by the Subtractive Three-colour Process
S. Papadakis *USP* 2,790,715

Cellulose ester film is first treated in a basic solution of a diazo sulphonate and the coupling agent for the blue component in a volatile solvent and exposed behind the negative of the blue component. It is then washed with alcoholic caustic soda to dissolve the diazo sulphonate and coupling agent which is untransformed by light and rinsed free of caustic soda. These operations are repeated for the yellow and red components in either order.
C.O.C.

Accelerator for Colour Developer Solutions
Novacolor Reprint Co. *USP* 2,794,741

When thiourea is used as accelerant in any known first developer used in reversal colour processing there is strong increase in the general fog level. This increase in fog is avoided by adding benzotriazole, *p*-nitrobenzimidazole or quinone in addition to the thiourea.
C.O.C.

Improving Colour Reproduction by means of Subsequent Masking when reproducing Photographic Subtractive Multicolour Images
Agfa *BP* 795,064

Inhibiting Discoloration of the Whites in Colour Photographs
General Aniline *USP* 2,788,274

Use of a 0·5–15·0% aqueous solution of a zinc salt as a final rinse inhibits yellowing of the whites when colour photographs are exposed to light, heat or high humidity.
C.O.C.

Increasing the Printability of Polyethylene Terephthalate Film (XIII p. 618)

Sensitometry of Multilayer Colour-photographic Materials. VI— Interrelation between Two Methods of Quantitative Evaluation of Colour Fields (XIV p. 619)

X— SIZING AND FINISHING

Factors affecting the Drying of Apparel Fabrics
R. Steele

I— Drying Behaviour
Text. Research J., **28**, 136–144 (Feb. 1958)

When fabrics of various natural and man-made fibres are dried under "drip-dry" conditions, the usual constant-rate and falling-rate periods are observed. The former time depends on the amount of water picked up by the fabric and is controlled by the fibre content and thickness of the fabric. With cotton and rayon fabrics it can be controlled to some extent by treatment with certain resins. For hydrophilic fabrics, the whole of the falling-rate period can be characterised by a drying coefficient based on the assumption that the area of free surface evaporation is the controlling factor and that this is proportional to the water content. With hydrophobic fabrics, the latter half of the falling-rate period is not consistent with this assumption, the rate being somewhat more rapid.

II— Capillary Size Distribution
Ibid., 144–147

The water-holding capacity of a fabric can be considered to be made up of the volume of a bundle of continuous capillaries of various radii, the volume of a non-continuous capillary system, and the sorption capacity of the fibres. A vertically suspended wet fabric develops a water concentration gradient from top to bottom which can be used to determine the pore size distribution of the bundle of continuous capillaries. The result is approximately a log normal distribution. For cotton printcloth, wool flannel, and nylon challis, the maxima of the radius frequency distributions were found to be 11·1, 19·3, and 9·3 μ. respectively.
S.B.D.

Comparative Mercerisation of Yarns made from Various Cottons
A. L. Murphy and C. F. Goldthwait
Text. Research J., **28**, 267–269 (March 1958)

The yarns were mercerised in several different ways to compare them under different conditions; very little difference was found among the cottons.
S.B.D.

Improved Light and Weather Resistance of Cotton resulting from Mercerisation
C. F. Goldthwait and H. M. Robinson
Text. Research J., **28**, 120–126 (Feb. 1958)

Comparative exposures of mercerised and unmercerised cotton to light and weathering brought out anomalous effects in the breakdown of cotton cellulose. There was less deterioration of the mercerised cotton as measured by breaking strength, but at the same time more degradation as determined by fluidity measurements, the latter indicating a reduction in the average D.P. of the cellulose. When ordinary unmercerised cotton has lost half its strength, the corresponding mercerised material can be expected to last for up to 33% longer before reaching that level.
S.B.D.

Fibrous Acetylation of Cotton Yarn
F. Bryant
Text. Research J., **28**, 180–181 (Feb. 1958)

The method consists in pretreatment in excess glacial acetic acid (2000 ml. per 4-oz. skein) for 60 min., and then immersion in a bath set with 666 ml. acetic anhydride, 1334 ml. toluene, and 0·8 ml. perchloric acid (70%) as catalyst for 35 min., followed by draining, drying, and then rinsing. The inclusion of the non-solvent prevents the cellulose acetate from dissolving in the bath and so gives fibrous acetylation. The process is found to give reserve effects when the yarn is subsequently incorporated in silk-rayon hose.
S.B.D.

Versatile Cotton in High-style Finishing
G. Heberlein and E. Weiss
Text. Research J., **28**, 227–233 (March 1958)

Methods used in actual plant operations for finishing cotton goods for special fashion effects are surveyed. By combining two or more treatments an unlimited number of fashion effects can be obtained. Cotton lends itself to a greater number of finishing methods than any other fibre, and this is demonstrated by a variety of high-grade finishes on fabrics.
S.B.D.

Acetal Cellulose Reactants
J. B. Irvine and B. H. Kress
Text. Research J., **28**, 148–158 (Feb. 1958)

Acetal cellulose reactants are a group of materials which produce non-chlorine-retentive dimensional control, a wide variety of handles from softness to permanent stiffness, and a range of crease resistances. Three types of acetal cellulose reactants are discussed. After a general description of their reactions with cellulose, an analysis of data obtained from laboratory evaluations of mill-processed fabrics is given.
S.B.D.

Dialdehydes as Cotton Cellulose Cross-linkers
M. D. Hurwitz and L. E. Conlon
Text. Research J., **28**, 257–262 (March 1958)

Formaldehyde is an effective cross-linking agent, but its use is accompanied by high strength loss in cotton. Work with the dialdehydes— glyoxal (C_2), glutaraldehyde (C_5), and α-hydroxyadipaldehyde (C_6) yielded crease recovery–strength relationships similar or identical to formaldehyde. The finding is consistent with the known chemistry of the formaldehyde–cellulose reaction in that the acetal linkage controls the length of the cross-link.
S.B.D.

Application of Resin Finishes to Cotton Garments using Dry-cleaning Plant Equipment
R. T. Graham, F. Loibl, and J. R. Wiebush
Text. Research J., **28**, 252–256 (March 1958)

The object of the work was to impart wrinkle resistance and desirable permanent creases to cotton garments. A tentative procedure for the resin treatment was devised and found effective on many weaves and weights of cotton fabric. The equipment used included a centrifuge, steam press or electric iron, hot-air drying cabinet, and a tumble drier.
S.B.D.

Relation of the Effect of Resins on Light Fading and the Tendering Action of Photosensitive Vat Dyes
AATCC Piedmont Section
Amer. Dyestuff Rep., **47**, P 39–P 48 (27 Jan. 1958)

There is a correlation between the vat dyes which lose light fastness after resin treatment and those which cause phototendering of cellulose. The dyes give peroxides when exposed to light, and the loss in light fastness after resin treatment has to be counteracted by the addition of an

inhibitor (diallyl phthalate) to the resin bath. A C–O bond in the resin—

$$-CH_2OH \quad \text{or} \quad \begin{array}{c} O \\ -\overset{}{C}-\!-\overset{}{C}- \\ H \quad H \end{array}$$

seems to cause the fall in light fastness. The resin itself seems to be responsible, not the catalyst. Details of the experimental techniques are given. Further experiments are projected. P.T.S.

Durable Creasing of Wrinkle-resistant Cotton
J. D. Reid, R. M. Reinhardt, and R. M. H. Kullman
Text. Research J., **28**, 242–251 (March 1958)

Under proper conditions of resin application and cure, it is possible to produce a cotton garment which has excellent wash and wear characteristics. If a garment is made from a resin-treated wrinkle-resistant cotton, it is sometimes possible to introduce a reasonably durable crease into the garment by heat in the presence of catalyst and water, this process being termed recuring. Two possible mechanisms for the recuring process are suggested, and evidence is given in favour of each. Results for the recuring of both laboratory and commercial resin-treated cotton fabrics are reported. S.B.D.

Wearing and Washing Trials of "Non-iron" Shirts
Wäschereiforschung Krefeld e.V.
Textil Praxis, **13**, 393–396 (April 1958)

A sample of 21 "non-iron" shirts of different manufacture was examined. The degree of whiteness and the freedom from creasing were assessed after the shirts had been washed and worn for 1, 3, 5, and 10 days. It was concluded that in the majority of cases the shirts still required light ironing. L.A.T.

Effect of Laundering on Chlorine Retention and Crease Resistance of Cotton finished with Dimethylolethyleneurea
O. C. Bacon, J. E. Smith, and L. E. Hughes
Amer. Dyestuff Rep., **47**, P 259–P 262 and P 272 (21 April 1958)

Fabrics which will be commercially laundered must be made crease-resistant with a resin which is not damaged by chlorine retention even after alkaline washing at high temperature or acid scouring. The finish on fabrics which will be washed at home need not be resistant to acid scouring. Dimethylolethyleneurea (DMEU) is a promising crease-resistant finish where no acid scouring is used. The curing catalyst for the resin which gives the best resistance to damage by chlorine retention is zinc nitrate. Magnesium chloride and alkylolamine hydrochloride are less good catalysts from this point of view, in this order. The discoloration of white fabric is least with magnesium chloride, greatest with zinc nitrate, and intermediate with the alkylolamine hydrochloride. Details of the experimental test methods are given. P.T.S.

Formaldehyde Fumes from Resin-treated Fabrics in Retail Stores
J. F. Mateson
Canadian Textile J., **74**, 73–77 (15 Nov. 1957)

The problem of formaldehyde fumes in air-conditioning retail stores is now becoming prevalent, for example, in New York. Air-conditioning systems in which the major part of the conditioned air is recirculated lead to a slow increase in formaldehyde concentration to levels at which it is readily perceptible and irritant to occupants. The formaldehyde arises from slow degradation of the urea– or melamine–formaldehyde finishes applied to textiles, owing to inadequate aftertreatment by washing and neutralisation or the use of treating resins of insufficient stability. The situation has arisen as a result of continuous efforts by manufacturers to reduce production costs and is aggravated by the large fluctuations in temperature and humidity between day and night when air-conditioning systems are employed. E.C.

Effect of Silicone Softeners on Resin-treated Cottons
B. G. Simpson
Amer. Dyestuff Rep., **46**, P 991–P 998 (30 Dec. 1957); *Text. Research J.*, **28**, 170–179 (Feb. 1958)

Application of thermosetting resins to cotton to impart a wash-and-wear finish reduces the strength of the cloth and adversely affects its handle. Application of a lubricant restores to some extent the strength and the handle. In this paper the use of silicone softeners (or lubricants) is compared with that of other anionic, cationic, and non-ionic softeners. The tear strength and the wrinkle recovery are higher after treatment with a silicone softener than with the other softeners, but the tensile strength and the flex abrasion resistance are slightly lower. When polymerised on the fibre the silicone softener is much more durable than any other softener. About half as much silicone is required for softening as for waterproofing. Silicones are good sewing lubricants. The experiments were carried out on mercerised cotton. P.T.S.

Rotproofing of Textiles
E. Higgins
J. Textile Inst., **49**, P 9–P 16 (Jan. 1958)

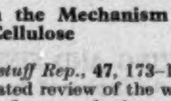
An entertaining account of the general principles of microbiology relevant to textile rotproofing. A textile fabric has to resist attacks from hundreds of types of microbes in unpredictable combinations which vary with every change of conditions— temperature, humidity, pH— and tests in the laboratory (especially those using pure cultures) are often completely inadequate to predict the efficiency of a rotproofing treatment in use. Some preliminary results with an artificial soil-like medium are presented, and the publication of further results is promised. P.T.S.

Oil- and Water-repellent Treatments for Cotton with Fluorochemicals
L. Segal, F. J. Philips, L. Loeb, and R. L. Clayton
Text. Research J., **28**, 233–241 (March 1958)

The chromium complexes of perfluoromonocarboxylic acids were used in conjunction with acrylic emulsions. These were employed in separate operations as well as combined with the complexes into one emulsion. The acids were applied to fabrics after impregnation with a mordant to bind them to the fibre. Data are presented to show the relative effectiveness of the repellency treatments as governed by the fabric construction. S.B.D.

Recent Work on the Mechanism of the Thermal Degradation of Cellulose
R. C. Laible
Amer. Dyestuff Rep., **47**, 173–178 (24 March 1958)

A very concentrated review of the work going on in the United States on flame and glow resistance imparted chemically to cellulose, and on the thermal degradation of cellulose. Cellulose fabrics treated with flame retardants produce less tar than do untreated fabrics when burnt. The ratio of tar to solid residue produced is some measure of the efficiency of the flame retarder. The ratio of CO to CO_2 produced in burning also measures flameproofing efficiency— treated fabrics liberate several times more CO. Inorganic substances are effective flame retarders: they can be held on to the fabric by binders, they may be chemically combined, or, for example, a phosphorus polymer may be applied to the cloth. Friedel-Crafts catalysts ($ZnCl_2$, $TiCl_4$, etc.) are flame retardants, and a theory of their mechanism is quoted, which involves a "dehydration" of the cellulose units to give water and carbon. Supporting this theory is the experimental fact that treated fabrics produce more water during pyrolysis than do untreated fabrics. Treated fabrics degrade more rapidly than untreated fabrics, and it is known that normally flammable vapours are sometimes not flammable at high concentrations. Fundamental work showed that laevoglucosan—



was probably the source of combustible products in cellulose degradation, and oxidised cellulose (in which some of the anhydroglucose units of cellulose are modified, so that *laevo*glucose cannot be formed) is less flammable than untreated cellulose. Results using radio-iodine to detect radicals formed in cellulose degradation could not be interpreted rigorously because of exchange reactions which made them equivocal. Gas chromatography has been used

with more success to detect products of pyrolysis in N_2, and work in O_2 is suggested. The CH_2OH group of anhydroglucose units in cellulose can be modified to give flame resistance. Replacing it by $CH_2{\cdot}O{\cdot}SO_2{\cdot}CH_3$ increases flame resistance, and the treatment does not affect other properties adversely. Replacement by $CH_2{\cdot}$Hal imparts flame resistance in the order: I > Br > Cl > F. Replacement by $CH_2{\cdot}O{\cdot}PO(OC_2H_5)_2$ increases glow resistance as well. Work on practicable methods of application produced a flameproofing treatment which can be applied in 5 min. More work on application is suggested, and the need for further research into the gaseous products of combustion is pointed out. 36 references. P.T.S.

Metallisation of Fabrics
S. Plum
Silk and Rayon, **32**, 378–383 (April 1958)
A review of techniques and equipment for the vacuum deposition of metals, particularly on cloth and polymer films. A.J.

PATENTS

Imparting Affinity for Basic Dyes to Cellulose
ICI *BP* 794,180
Cellulose is treated in aqueous alkali with a compound (other than a dye) containing at least one 1:3:5-triazine ring whose C atoms carry as substituents two halogen atoms and a residue of a primary or secondary amine bound via its N atom and containing at least one negatively charged solubilising group. Thus spun viscose rayon cloth is padded with an aqueous solution containing 3% of the primary condensate of 1 mol. of cyanuric chloride and 1 mol. of *p*-amino benzene sulphonic acid and 3% of NaOH. The treated cloth is dried, rinsed and again dried before being printed with dyes solubilised by cationic groups. Much strong and more level prints are obtained than are obtained in the untreated cloth.
BP 794,181
Similar compounds are used whose C atoms are free from lipophile residues, e.g. the condensate of 2 mol. of 2-β-hydroxyethylamine-4:6-dichloro-1:3:5-triazine and 1 mol. of *pp'*-diaminostilbene disulphonic acid. C.O.C.

Treating Cyanoethylated Cotton with Amines
Institute of Textile Technology *USP* 2,787,516
USP 2,793,930
Cyanoethylated cotton, e.g. that produced by treating cotton with NaOH and then with acrylonitrile, is treated with a liquid amine or an aqueous solution of an amine. This confers increased tensile strength, elongation and abrasion resistance. 3-Substituted propylamines, e.g. 3-*iso*propoxypropylamine, are very effective amines for use in this process. C.O.C.

Sizing Acrylic Yarns
National Starch Products *USP* 2,788,293
Starch, preferably a starch derivative containing acidic groups, is used together with a stannic, stannous, ceric, chromic, chromous, bismuth or cuprous salt, to give efficient and cheap sizing. C.O.C.

Stabilising Nylon to Ultraviolet Radiation
Sherwin-Williams Co. *USP* 2,790,734
Nylon which has been treated with a Cu salt of an organic carboxylic acid and then cured at 250–500°F. for $\ngtr$ 10 min. is given protection against loss of tensile strength on exposure to ultraviolet radiation. Preferably the copper salt is used in conjunction with a resinous material as binding agent. C.O.C.

Giving Textiles Dimensional Stability and Ability to Recover from Creasing
Shell Development Co. *USP* 2,794,754
The materials are impregnated with an aqueous mixture of a compound containing several vic-epoxy groups, an organic compound containing at least one XCH_2OH group (X = N, C atom of an aromatic ring or an aliphatic or *cyclo*aliphatic C atom attached to a > CO group) and an epoxy curing agent and then baked. The handle is not affected and the finished material is not chlorine-retentive. C.O.C.

Crinkled Nylon Yarn
Scott & Williams *BP* 792,110
Nylon or other thermoplastic yarn is continuously crinkled by passage through a modified circular knitting machine. Sliders replace the needles and operate in conjunction with sinkers to produce a zig-zag configuration of the yarn whilst heat is applied. The yarn is cooled by an air-blast on leaving the machine and wound on to package. G.E.K.

Stiff and Flame-resistant Finish for Nylon
Monsanto *USP* 2,795,513
The material is treated with a composition comprising (1) a water soluble, fully methylolated heterocyclic compound having a saturated ring containing only C atoms to one of which are attached 2 NCH_2OH groups, there being at least one O or S atom also attached to that C atom, e.g. dimethylol ethylene urea, (2) thiourea, and (3) a latent catalyst and then baked. The stiff flame-resistant finish is fast to washing and dry-cleaning. C.O.C.

Rendering Cellulosic Materials Resistant to Shrinking
Battelle Development Corpn. *BP* 794,764
Cellulosic textiles are treated with a polysulphate or polysulphonate of an alkyl or aralkyl hydrocarbon or with an oxygenated derivative of such a compound. Thus cotton cloth was immersed for 60 sec. at 210°F. in an aqueous solution containing NaOH (15·7% by wt.) and 2-hydroxy-1:3-propane disodium sulphonate (13·5), washed and dried. The treated cloth retained its dimensions even on repeated laundering. C.O.C.

Improving the Fastness to Washing of Disperse Dyes on Acetate Rayon
Celanese Corpn. of America *BP* 794,179
Dyeings of disperse dyes containing a reactive group on cellulose acetate have their fastness to washing improved by heating them with an amidogen-formaldehyde condensate and HCHO. Thus a 50/50 acetate/viscose rayon blend is dyed brown with the blue disperse dye 1:8-dihydroxy-4-*p*-(β-hydroxyethyl)anilido-5-nitroanthraquinone, the orange disperse dye 4-nitro-2:6-dichlorophenyl-azo-4'-bis (β-hydroxyethyl)-amino-2'-chlorobenzene, the red disperse dye 4-nitro-2-methoxyphenylazo-4'-bis (β-hydroxyethyl)-amino-2'-acetylaminobenzene, Resofix Blue GL, Cuprofix Yellow GL and Resofix Rubine BLN. The dry dyed material is then padded with dimethylol urea, *N*:*N'*-di-(hydroxymethyl)-ethylene urea, Cuprofix 47, Catalyst G–8 (a mixture of an organic amine hydrochloride and HCHO) and a cationic softening agent. It is then dried and baked. C.O.C.

Radiation-resistant Fabric
J. M. Stertz *USP* 2,788,291
Fabric is rendered resistant to radioactive particles of air, dust, water or fire by first rendering it water-repellent and flame-resistant and then treating it with a lead soln., e.g. with a solution containing approx. 80% Pb, 12% SO_2, and 8% Al by wt., and as bonding agent glycovinyl resinate. The solution is made by melting the Pb and then adding the SO_2, Al, and resin; it remains sufficiently liquid for treatment to be carried out at 60–80°F. C.O.C.

Crimped Nylon Hose
British Nylon Spinners *BP* 791,960
False-twist crimped nylon hose are pressure steamed in an unboarded, relaxed condition, before dyeing, and finally boarded at a temperature lower than that of the initial steaming. G.E.K.

Preventing Stove Staining in the Manufacture of Linoleum
Armstrong Cork Co. *BP* 795,176
When making linoleum by applying a linoleum composition to a base containing proteinaceous material, stove staining is prevented by first treating the proteinaceous material with a tanning agent. C.O.C.

Imparting Affinity for Acid Dyes to Cellulose Acetate Fibres (VI p. 608)
Reactivity of Cellulose (XI below)
Swelling and Cavity Formation in Secondary Cellulose Acetate (XI p. 616)
Application of Yarn Hairiness Measurement Techniques to the Control of Gassing or Singeing (XIV p. 619)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Reactivity of Cellulose
A. R. Urquhart
Text. Research J., **28**, 159–169 (Feb. 1958)
The chemical finishing of cotton entails applying to it materials that react either with the cellulose or with each

other after they have been adsorbed by the cellulose. In both cases the cellulose must be readily accessible and in a reactive condition. The paper discusses the reasons underlying the great differences of reactivity that can be encountered among samples of cellulose that appear to be identical chemically and differ only in physical form or structure. Moisture absorption studies exemplify these differences and build up a theory that goes far to explain them in terms of variability of the availability of the reacting groups in the cellulose molecule. Thus purified cotton is in general in a state of low reactivity and regenerated cellulose in one of high reactivity, while mercerised cotton occupies an intermediate position. This picture is satisfactory, for most reactions, but in some cases it is completely reversed, with ordinary cotton having high reactivity and the regenerated cellulose low reactivity, mercerised cotton again being intermediate. The cause of this is discussed in the light of more recent work.

S.B.D.

Calculation of Intermolecular Forces by means of Density Measurements

A. Sippel

Das Papier, **12**, 86–89 (March 1958)

A contribution to the problem of the range of intermolecular forces of cellulose in which a new interpretation is given of the process of diluting the solution. A shell-like structure of the solvent is assumed. The pressure gradient, which is calculated from the shell densities or the compressibility of the solvent, leads to the law of attraction in relation to distance. For sucrose, used as a model for cellulose, it was found that a very large number of water molecules are attached fairly firmly to it. R.A.

Degradation of Cellulose in Air

H. G. Higgins

J. Polymer Sci., **28**, 645–648 (April 1958)

Degradation of cellulose in air at 250°c. is studied by infrared spectroscopic examination. The oxidative nature of the degradation is shown by development of C–O groups. The reaction quickly penetrates crystalline regions. A relatively stable structure in which carbonyl groups replace aldehyde groups develops after about 12 hr.

W.R.M.

Degradation of Cellulose in a Vacuum by Ultraviolet Radiation

J. H. Flynn, W. K. Wilson, and W. L. Morrow

Bur. Stand. J. Res., **60**, 229–233 (March 1958); *Research Paper* 2841

Cotton cellulose sheets were irradiated in a vacuum at 40°c. with radiation of wavelength 2537Å. Hydrogen, carbon monoxide, and carbon dioxide were evolved, the D.P. decreased, and CHO and COOH groups were produced. The rate of evolution of hydrogen suggests inhibition by a product. The rate of evolution of the carbon oxides increased slightly during irradiation. Quantum yields for hydrogen and the carbon oxides are given. A mechanism is proposed by which alcohol groups are converted to carbonyl groups with liberation of hydrogen.

W.R.M.

Comparative Reactivity of Hydroxyl Groups of Cellulose

V. Derevitskaya, M. Prokof'eva, and Z. Rogovin

J. Gen. Chem. U.S.S.R., **28**, 716–718 (March 1958)

V—Distribution of Methoxyl Groups in partially Methylated Cellulose obtained in Alkaline Media with Different Alkali Concentrations

Methylation of cellulose by methyl iodide in alkaline media (between 8 and 40%) is investigated. Between 13 and 40% alkali the distribution of methoxyl groups in methylcellulose changes with the alkali concn. The formation of methylated cellulose occurs selectively through reaction of the secondary alcoholic groups, the selectivity max. being at 40% alkali. A comparison of the methylation of unwashed and washed alkali cellulose shows higher γ values (169–179) in the former case, but only 88–105 in the latter.

VI—Distribution of Methoxyl Groups in the Products of Methylation of Cellulose Sodium Alcoholate

Ibid., 718–721

Partially methylated cellulose obtained in the above manner is formed by methylation of the secondary alcoholic groups. When this occurs, γ values of up to 100 are obtained. On the other hand, γ values > 100 involve the reaction in addition of the primary alcoholic groups.

G.J.K.

Sensitivity to Alkalis of the 1–4 Acetal Linkages of Oxycelluloses

P. Rochas

Bull. Inst. Text. France (70), 21–32 (Oct. 1957)

The author shows that the sensitivity of oxycelluloses to alkalis is linked with the presence of a carbonyl group in positions 2 or 3 of the glucopyranose ring, irrespective of whether the 2–3 carbon–carbon bond is intact or not. Thus the sensitivity to alkalis of oxycelluloses of the 2:3-dialdehyde type is eliminated by both sodium chlorite and potassium borohydride, but not by diazomethane, whereas in the case of oxycelluloses containing both 2:3-dialdehyde functions and 2-ketone functions sodium chlorite decreases the alkali sensitivity without eliminating it, whilst potassium borohydride eliminates it completely. Various mechanisms are suggested to account for the effect of the carbonyl groups on the 1–4 and 1–5 (intra-annular) acetal linkages. J.C.F.

Oxidation Reactions of Cellulose studied by Infrared Spectroscopy

I. N. Ermolenko, R. G. Zhbankov, V. I. Ivanov, N. Ya. Lenshina, and V. S. Ivanova

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 249–251 (Feb. 1958)

The oxidation of cellulose by NO_2 is shown, on the basis of the absorption band at 7 μ., to result in simultaneous formation of carboxylic and non-carboxylic groups at the 6-carbon atom. The action of $NaIO_4$ on cellulose also results in partial oxidation at the same atom. The presence of the absorption band at 11 μ., characteristic of the aldehyde group, indicates the formation of dialdehydocellulose when $NaClO_2$ is the oxidant. G.J.K.

Kinetics of the Oxidation of Cellulose with Nitrogen Dioxide from the Absorption Spectra of the Products

I. N. Ermolenko and M. M. Pavlyuchenko

J. Gen. Chem. U.S.S.R., **28**, 722–728 (March 1958)

The authors claim that their earlier statement that cellulose nitrite is formed in the NO_2 oxidation of cellulose is confirmed by spectroscopic evidence. The kinetics of this oxidation at 31°c., obtained from the spectra, show a relation between the max. amount of nitrite in the oxidation product and the rate of accumulation of the carboxyl groups. The reaction has been investigated at 4 different temp.— 25, 31, 44, and 65°c. Increasing temp. results in a decrease in the formation of (*a*) carboxyl groups and (*b*) cellulose nitrite, longer time being required to attain max. cellulose nitrite concn. A comprehensive bibliography of recent work in this field is given. G.J.K.

Osmotic Measurements on Organic Xanthates

J. Schurz and W. Zimmerl

Svensk Papperstidning, **61**, 157–161 (31 March 1958)

Using an osmometer developed by Breitenbach and Forster from an older type by Hellfritz and Zimm-Myerson, four concentrations of each xanthate (diethylacetamidocellulose xanthates) in methyl sulphoxide were studied. The values of D.P. were found to be 2–3 times those obtained by usual methods, falling to normal value in 10–14 days. The effect could be explained by aggregation of the xanthate solution, followed by dissociation. This does not agree, however, with viscometry measurements, and it is therefore suggested that association processes are responsible for the phenomenon. R.A.

Swelling and Cavity Formation in Secondary Cellulose Acetate

R. Jeffries

I—Mechanism of Cavity Formation

J. Textile Inst., **49**, T 192–T 213 (April 1958)

An investigation of the variables influencing the delustring of secondary cellulose acetate (and other polymers — polystyrene, ethylcellulose, polymethyl methacrylate, cellulose nitrate) by cavity formation in solvent–non-solvent mixtures or partial solvents. Swelling precedes cavity formation. The rate of swelling can be followed, the amount of polymer dissolved can be measured, the amount of solvent absorbed can also be measured. These can be correlated with the molecular weight of the polymer. Suitable solvent–non-solvent mixtures are— phenol–water, acetone–water, acetone–carbon tetrachloride, phenol–alcohol. The effect of wetting agent was examined. The

cavities formed (it is concluded from the mass of experimental data) contain an internal solution of low-mol.wt. polymer. They empty during drying and form irregularities in the fibres which scatter light. It is difficult to achieve reproducible results with a given delustring process—the results vary from source to source and even from batch to batch. It is difficult to delustre highly oriented samples. Delustring in this way considerably weakens the fibre. Flaws in the original structure probably form the nuclei for cavities.

II—Effects of Surface-active Agents and Salts
Ibid., T 214–T 222

Addition of surface-active agents to phenol–water increases the degree of delustring attainable as measured by optical density. 12 surface-active agents were used, and a mechanism is advanced for their action within the polymer, not on its surface. Addition of inorganic salts to the aqueous phenol invariably causes reduced delustring at 100°c., the temperature usually employed for delustring. The effect is said to be due to a decrease in the activity, and thus the absorption, of the water. At lower temperatures, there is sometimes enhanced delustring owing to absorption of inorganic ions by the cellulose acetate. The enhancement is greater with univalent than with bivalent ions. This enhancement competes with the lowering of the activity of the water mentioned before.
P.T.S.

PATENT

Coated Boxboard
Lowe Paper Co. *BP* 795,175

A hydrophobic, non-oleaginous thermoplastic basecoat is first bonded to a substrate after which a mineral pigment-adhesive topcoat is applied in aqueous suspension and dried. The coatings are then united by fusion at a temperature high enough to soften the thermoplastic material of the basecoat. The boxboard is preferably composed of reprocessed pulp and the thermoplastic material may be any of the usual polymers or copolymers of ethylene, acrylonitrile, butadiene, polyvinyl chloride, cellulose acetate and polyvinyl butyral. R.A.

Aqueous Dispersion of Styrene–Butadiene Copolymers—Coatings for Washable Wallpapers, Foils, etc. (XIII p. 618)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Periodic Deformation of Collagen in Solutions of Electrolytes and Tanning Agents
A. G. Pasÿnskiĭ and A. M. Tongur
Colloid J. U.S.S.R., **19**, 483–489 (July–Aug. 1957)

The dependence of the periodic deformation of leather collagen in water and in various electrolytes (2M-NaCNS, KCNS, LiCl, 5M. urea, 25% $(NH_4)_2SO_4$) upon frequency is studied in the range 1–1,000 cycles/min. It is shown that at temp. below the shrinkage point collagen exhibits only small relaxation periods, behaving as an elastic protein gel. Chrome-tanned leather, on the other hand, possesses a large relaxation period. Use has been made of this phenomenon in accelerating the penetration of the tanning agent into the collagen by subjecting chrome-tanned leather in tanning solutions to periodic compressions of a frequency corresponding to the large period of relaxation.
G.J.K.

Determination of *N*-Terminal Amino Acids of Collagen by the 2:4-Dinitrofluorobenzene Method
C. Deasy
J. Amer. Leather Chem. Assocn., **53**, 203–208 (April 1958)

The exact nature of the chemical changes that occur when collagen is converted into gelatin is not known, but as the degradation of the collagen molecule is probably involved it is very likely that the *N*-terminal amino acids of collagen should be amongst those of gelatin. Serine, aspartic acid, and glycine have been found, by means of a modification of the 2:4-dinitrofluorobenzene method, to be *N*-terminal acids of ox-hide collagen; the same three have been reported elsewhere in the literature as *N*-terminal acids of gelatin. J.W.D.

Modification of the Protein Molecule. II—Addition of Amino Acids and Peptides to the Carboxyl Groups in the Protein Molecule
K. T. Poroshin
Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 236–237 (Feb. 1958)

Derivatives of casein and egg albumin are obtained by adding esters of amino acid, e.g. those of glycine, DL-alanine, diglycine, and DL-alanyldiglycine, to the carboxylic acid groups of these proteins. G.J.K.

Complex Formation between Groundnut Skin Pigments and Conarachin II
H. M. R. McDavid and W. E. F. Naismith
Nature, **181**, 480–481 (15 Feb. 1958)

Sedimentation velocity diagrams of various extracts of groundnut show a fast component (s°_{20} = 32 S.) associated with the red colour of the skins. Treatment with ethyl methyl ketone to extract vegetable tannins causes loss of colour and the replacement of the s_{32} compound by what appears to be conarachin II, suggesting that the s_{32} component was a complex between conarachin II and skin pigments. A.J.

XIII—RUBBER; RESINS; PLASTICS

Control of Spherulitic Texture in 6,6-Nylon
C. R. Lindegren and M. I. Kohan
J. Polymer Sci., **28**, 615–616 (April 1958)

The spherulitic structure of 6,6-nylon can be varied by control of the melt temperature. Slow cooling from a maximum temperature of 278°c. results in large spherulites, while cooling from a max. temp. of 269°c. gives much smaller spherulites. The number of active spherulite nuclei decreases with increasing melt temperature and increasing time in the melt. W.R.M.

Copolymerisation of ε-Caprolactam with 2-Vinylpyridine or 2-Methyl-6-vinylpyridine in Bulk and in Solution
J. Tomaszewski and S. Chrzczonowicz
Zesz. Nauk. Politech. Łódz, (18) (*Chemia* 6), 57–60 (1957)

The polyamide obtained from the polymerisation of the ε-caprolactam in boiling xylene catalyses its polymerisation in the bulk and its copolymerisation with 2-vinylpyridine and 2-methyl-6-vinylpyridine. The copolymerisation is assisted by metallic Na and anhydrous NaOH, and must be carried out in an inert atmosphere of CO_2 at the b.p. of ε-caprolactam. It is complete after 15 min. It is possible to carry out the reaction under pressure in 5–6 hr. at 260–270°c. or in boiling xylene. The copolymers so produced show fibre-forming properties and greater solubility in H_2SO_4 than the homogeneous polyamide. T.Z.W.

Hetero-chain Polyamides

VI—Synthesis of Polyamides and Polyamide Esters by Aminolysis of Polyesters
V. V. Korshak, T. M. Frunze, and V. F. Petrova
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, 217–220 (Feb. 1958)

The polyester obtained from the polycondensation of ethylene glycol with sebacic acid undergoes aminolysis with hexamethylenediamine, forming either polyamido-esters or polyamides depending on the ratio of the initial reactants.

VII—Pyrolytic Decomposition of Polyhexamethyleneadipamide
V. V. Korshak, G. L. Slonimskiĭ, and E. S. Krongauz
Ibid., 221–226

The effect of heating in an inert atmosphere on the mol. wt. of polyhexamethyleneadipamide is investigated. It is found that, irrespective of the D.P. of the starting material, decomposition of the molten polyamide produces a substance whose mol. wt. depends only upon the temp. The nature of the end-groups of the polyamide has no effect upon the establishment of the equilibrium value of the mol. wt. during heating in an inert atmosphere. The results are explained on the basis of a decomposition–recombination equilibrium mechanism. G.J.K.

Effect of Impurities in Melamine on its Condensation with Formaldehyde
L. M. Pesin, V. N. Katrelev, A. E. Zarubitskiĭ, and F. E. Segalevich
J. Appl. Chem. U.S.S.R., **31**, 146–148 (Jan. 1958)
The alkali-soluble organic impurities in melamine, consisting mainly of ammeline and ammelide, have a detrimental influence on its condensation with formaldehyde. They increase greatly the speed of the polymerisation, which greatly decreases the quality of the product. T.Z.W.

PATENTS

Colouring the Surface of Polyethylene Amber
Allied Chemical & Dye Corpn. *USP* 2,793,964
The polyethylene is treated with fluorosulphonic acid or with sulphur trioxide and tetrachloroethylene; it becomes permanently brown with a greenish cast. On washing with alkali the colour changes to golden brown or amber. C.O.C.

Rendering Polyester Films Receptive of Printing Inks, Coating Compositions, etc.
Minnesota Mining & Manufacturing Co. *USP* 2,786,778
Polyester film after being treated with silicon monoxide vapour is highly receptive to printing inks, coating compositions, etc. C.O.C.

Increasing the Printability of Polyethylene Terephthalate Film
ICI *BP* 796,341
Polyethylene terephthalate film that has been molecularly oriented by drawing but has not been heat set may have its bonding properties improved, particularly the adhesion of ink impressions, by subjecting its surface to high voltage electric stress accompanied by corona discharge. The other desirable properties of the film are not deleteriously affected and the improvement obtained is not removed by heat setting. C.O.C.

Pigmented Organic Plastics
B. F. Goodrich Co. *USP* 2,793,195
The solid organic plastic and pigment are treated in a ball, pebble or colloid mill with water, a liquid organic water-miscible organic solvent for the plastic and a small amount of a liquid water-immiscible organic solvent for the plastic, until the desired degree of dispersion is obtained. This is a simple and cheap means of preparing master colour batches. C.O.C.

Aqueous Dispersion of Styrene–Butadiene Copolymers— Coatings for Washable Wallpapers, Foils, etc.
Chemische Werke Hüls *BP* 794,837
Aqueous dispersion of copolymers of styrene (70–99% by wt.) and butadiene (30–1) which contain a volatile organic solvent (1–10% on wt. of the copolymer) yield excellent coatings at room temperature without use of pressure. They have varied uses, e.g. as coatings to produce washable wallpapers, production of foils, etc. C.O.C.

XIV— ANALYSIS; TESTING; APPARATUS

Determination of Calcium in Spent Sulphite Liquor
O. Franzon, G. Ivarsson, and O. Samuelson
Svensk Papperstidning, **61**, 165–166 (31 March 1958)
An aliquot containing not more than 4 m-mol. of Ca is passed through a cation-exchange resin. All traces of spent liquor are carefully removed by washing with deionised water. 25 ml. of the diluted buffer is passed through, after which the column is eluted with 0·1 M. ethylenediaminetetra-acetic acid (E.D.T.A.), containing 10% by vol. of Buffer 10 (70 g. NH_4Cl + 570 ml. conc. NH_4OH (sp.gr. = 0·90) per litre). The bed is finally washed with diluted buffer, and the excess E.D.T.A. is titrated with 0·05 or 0·01 M-$MgSO_4$ using a mixed indicator comprising Eriochrome Black T and Methyl Red. The colour change is from green to pink, turning grey at the equivalent point. R.A.

Chromatographic Examination of Oils with Dyed Filter Paper
D. W. Poxon
J. Oil & Col. Chem. Assocn., **41**, 291–300 (April 1958)
Fixed and essential oils may be identified and the ageing properties of the former determined by chromatography based on suitable dyes and filter papers cut to a specific shape. The method appears promising as a means of identification for other organic cpd. of high mol. wt., e.g. surfactants. J.W.D.

Quantitative Determination of Antioxidants— III
J. W. H. Zijp
Rec. Trav. chim., **77**, 129–132 (Feb. 1958)
Detailed procedures are given for the colorimetric determination of (I) *p*-(*p'*-tolylsulphonylaminophenyl)-phenyl-*p*-toluidine, (II) *NN'*-di-*o*-tolylethylenediamine, and (III) hydroquinone monobenzyl ether. I, when treated with bis-2:4-dichlorobenzoyl peroxide in benzene, gives a reddish product which has constant absorption after 30 min. at room temperature. II and III, in aqueous ethanol, are heated with diazotised sulphanilic acid; II gives maximum colour in 20 min. in neutral solution; III is treated in alkaline solution for 15 min. Preliminary calibration is necessary. E.V.T.

Measurement of Covering Power of White Pigments
H. H. Weber
Farbe und Lack, **63**, 586–594 (1957):
Chem. Abs., **52**, 6808 (25 April 1958)
A discussion of the relationship, for light of a given wavelength, between the intrinsic reflectivity R_∞ (reflection for infinite paint thickness), the reflection constant S, the paint thickness X, and the reflection R_0 from a painted black surface. The covering power of a paint is defined as the reciprocal thickness $1/X_D$ of a paint on a black surface required to make R_0 = 0·98R. The dependence of S and $1/X_D$ on the volume concentration of the pigment is linear at first, but rises more slowly for heavily pigmented paint. The dependence of S and $1/X_D$ on particle size is illustrated by work to ZnS, the experimental (C.I. Pigment White 7) data being in agreement with the Mie theory. Data are also given for $BaSO_4$ (C.I. 77120), white lead (C.I. Pigment White 1), rutile and anatase (C.I. Pigment White 6). C.O.C.

Comparison of Fabric Tear Tests
R. Steele
Amer. Dyestuff Rep., **47**, 143–145 (10 March 1958)
The efficiency of tear tests has been examined in relation to the effect of finishes on tear strength. Tear strength of a fabric is related to its deformability and the ultimate tensile strength of its component threads; and applied finishes can affect both these properties. Six methods of tear testing are compared. Single-rip tear testing permits the greatest distortion of the fabric and so is very sensitive to treatments which affect the ease of distortion of a fabric, e.g. surface lubricants. The Elmendorf, wing-rip, and trapezoidal tests are unaffected by such treatments. The pin test and double-rip tongue tests seem to be intermediate in their sensitivity to lubricants. P.T.S.

Ultraviolet Reflectance Curves of Textile Materials. I— Fibre Identification
G. Thompson, C. A. Lermond, and R. A. Kenney
Amer. Dyestuff Rep., **47**, P 267–P 272 (21 April 1958)
It is believed by the authors that ultraviolet reflectance characteristics are as much a fundamental property of textile fibres as tenacity, specific gravity, melting point, etc. The reflectance curves of cellulosic fibres, protein fibres, polyamides, polyesters, and acrylonitrile and vinyl fibres are given in the paper, and the effects of delustrants and different bleaches can be distinguished. Provided that the fibres are not dyed or finished, the method provides a quick means of identification. Further work is promised on the effects on the ultraviolet spectra of fibres of various finishes and exposure to light and heat. P.T.S.

Estimation of Cortical Components of various Wools
W. J. Thorsen
Text. Research J., **28**, 185–197 (March 1958)
Widely varying wools were stained with various reagents chosen to give high contrast between ortho- and para-cortical segments. The dark stain produced by sodium plumbite, selective for the paracortex, was measured and found to increase with diameter and with the age at shearing. Wool fibres which had been elongated or supercontracted also showed increased staining, reflecting mechanical damage. The same wools were stained with acid nickelous nitrite, selective for the orthocortex, and it was verified that the order of staining was the reverse of that with plumbite. Solubility in an aqueous soln. of urea and monothioglycol is shown to increase regularly with the degree of staining with the nickel soln. Bromine water also

preferentially stained the orthocortex. Paralleling the staining behaviour, the stress in wool at 30% wet extension increases generally with diameter as well as with increasing maturity of the sheep. S.B.D.

Removal of Cotton or Viscose Rayon in the Analysis of Fabrics containing Nylon Blends
W. S. Sondhelm and H. Webster
J. Textile Inst., **49**, T 171–T 172 (March 1958)

The arrangement of the nylon fibres after the cellulosic component has been removed from a blend can be maintained if the area over which the cellulose component is to be removed does not greatly exceed the length of the nylon staple used in the blended yarn. In the method described the cellulosic component outside the area of removal is protected from the action of the cuprammonium solvent by pressing it into polyethylene, from which a circle has been cut for the test, at 120°C. P.T.S.

Identification of Unknown Synthetic Fibres
S. G. Smith
Amer. Dyestuff Rep., **47**, 141–142 and 145 (10 March 1958)

Optical properties in four liquids (1-bromonaphthalene, monochlorobenzene, methyl salicylate, and mineral oil of $n = 1{\cdot}470$) are used to distinguish between Dacron, nylon, viscose rayon, secondary cellulose acetate, Arnel, Darvan, Acrilan, Creslan, Orlon, Zefran, polyvinyl chloride, alginate rayon, dynel, Verel, Vicara, and Saran. The Becke line is a fine bright line of light along the edge of a fibre which moves either into or away from the fibre when the microscope is focussed, depending on the relative refractive indices of fibre and immersing liquid. It is observed through a cap analyser both parallel and perpendicular to the fibre length. Movement of the Becke line in the four liquids is observed. The appearance of the fibre (coloured or colourless) using crossed Nicol prisms is also observed, and these tests allow the unknown fibre to be put in one of five categories. Definite characterisation of the fibre follows after confirmatory tests involving solubility of the fibre, and appearance of its cross-section. All the tests are carried out under the microscope. P.T.S.

Evaluation of Test Methods for Milled Fabrics
AATCC Northern New England Section
Amer. Dyestuff Rep., **47**, P 149–P 154 (10 March 1958)

The problem was to relate the judgment of an expert on the quality of milling to some quantity measurable in the laboratory. To evaluate felting or milling it is necessary to measure not only the fibre randomness but the distribution of the felted fibres through the thickness of the cloth. Measurements of weight, thickness, compressibility, abrasion resistance, air permeability, fluid flow, and β-ray penetration vary with the degree of felting, but they are affected only by fibre randomness and not by the distribution of felted fibres through the cloth. Thus they cannot be used to measure the quality of the milling. Details of the tests are given. P.T.S.

Identification of Dye Classes on Dyed Natural and Synthetic Fibres
AATCC New York Section
Amer. Dyestuff Rep., **47**, P 9–P 17 (13 Jan. 1958)

The identification scheme is divided into six sections, each dealing with dyes for particular fibres— (i) cotton and rayon, (ii) wool and silk, (iii) secondary cellulose acetate and cellulose triacetate, (iv) 6- and 6,6-nylon, (v) acrylic fibres, and (vi) Dacron polyester fibre. In sections (iii)–(vi) tests are given for the fibre also, since this eliminates dyes not used to dye the particular fibre. Exact experimental details are given, and the importance of intelligent application of the tests is emphasised. P.T.S.

Lighting Factors to be considered in Textile Colour Matching
W. B. Reese
Amer. Dyestuff Rep., **47**, P 49–P 56 (27 Jan. 1958)

A popular account. Two illuminants are required for colour matching— the best are north sky daylight and horizon sunlight. P.T.S.

Prexa Apparatus for Washing and Milling Fastness Tests
Wullschleger & Schwarz
Dyer, **119**, 621 (25 April 1958)

Recommendations for carrying out mechanical wash fastness and milling tests on Prexa machines, manufactured by Wullschleger & Schwarz, Basle (cf. J.S.D.C., **73**, 357 (July 1957)). C.J.W.H.

Sensitometry of Multilayer Colour-photographic Materials. VI— Interrelation between Two Methods of Quantitative Evaluation of Colour Fields
Y. K. Vifanskiĭ, Y. M. Gorokhovskiĭ, and I. N. Konyushkova
Uspekhi Nauch. Fot., Akad. Nauk S.S.S.R., Otdel. khim. nauk, **5**, 127–144 (1957): *Chem. Abs.*, **52**, 6990 (10 May 1958)

The optical d. of a dye image is not exactly proportional to the concentration of the dye. Hence, ds. determined by measuring the dye concentration cannot be related to ds. determined spectrophotometrically by linear equations. A system of linear equations is given for the approximate calculation of effective ds. from dye concentrations and also from spectrophotometric data. C.O.C.

Application of Yarn Hairiness Measurement Techniques to the Control of Gassing or Singeing
A. Barella and M. Ruiz-Cuevas
J. Textile Inst., **49**, P 4–P 8 (Jan. 1958)

Fibres protruding from the core of a yarn may (i) be securely anchored, (ii) have both ends anchored and form loops, or (iii) be "wild" fibres insecurely wound about the core of the yarn. Two photoelectric regularity meters have been used to investigate the effects of singeing and wet processing on hairiness. The results show that the optimum speeds for singeing two cotton yarns are 400 and 600 metres/min. The effect of varying the temperature of the heating element has not been fully considered, but preliminary experiments show that its effect is small. The combined effects of boiling, bleaching, and sizing on hairiness are of a second order compared with the effect of singeing. Sizing lays protruding fibres close to the body of the yarn. P.T.S.

Devices and Techniques for use in Fungicide Testing
S. Shapiro
Amer. Dyestuff Rep., **47**, 73–78 (10 Feb. 1958)

The devices and the techniques described here make research into fungicides more efficient, reproducible, and safe, and less tedious. A cabinet for the horizontal drying of test strips of fabric impregnated with fungicide prevents the migration which would occur if the fabric were held vertical to dry. Impregnated fabrics should not be piled one upon another— simple storage trays prevent this and yet use the available space efficiently. A layer of vulcanised rubber on the jaws of a Scott tensile strength test machine prevents fabric samples slipping. A cutting die— in effect, a multiple cork-borer— simplifies the sampling of fabrics for chemical analysis. The fumes from organic solvents are sucked downwards, and not upwards as in a normal fume-cupboard, to minimise the risk to operatives. Standard samples for soil-burial tests are cut using a template and a finger-rule. Paint fungicides are evaluated by painting pieces of filter paper. Small squares are cut from the paper and inoculated with fungus. A rubber or metal stamp facilitates cutting the filter paper and estimation of the efficiency of the fungicide. A fabric with coloured threads woven in facilitates the cutting of samples for soil-burial tests. A template with regular openings cut in it makes it possible to estimate the proportion of a painted surface attacked by fungi. The size of the template openings necessary for a certain accuracy can be arrived at statistically. P.T.S.

Identification and Estimation of Phenolic Fungicides in Mildewproof Materials
C. L. Hilton
Text. Research J., **28**, 263–266 (March 1958)

An ultraviolet method of analysis has been developed. Accuracy and precision of ±3% have been obtained. The ultraviolet absorption spectra of twelve compounds (thirty commercial fungicides) are included. S.B.D.

Fire Accidents— the Contribution of Some Textiles
D. I. Lawson
Research, **11**, 126–133 (April 1958)

The flammability of apparel and household textiles makes an important contribution to accidents in the home. The rate of flame spread over fabrics is a reliable indication of the burning hazard, and work is in progress at the Fire Research Station, Boreham Wood, Hertfordshire to ascertain if there is any correlation between the severity of burns and the time taken by any fabric to burn 100 in. vertically. C.O.C.

Printability Studies on a Survey Series of Paperboards and Coated Papers
J. M. Fetsko

T.A.P.P.I., 41, 49–63 (Feb. 1958)

The reliability of various instruments is evaluated by laboratory proof-press results. Smoothness tests were made with the Bekk, Gurley, Sheffield, Bendtsen, and Chapman testers, surface absorbency and gloss by the Hull wipe test, and gloss holdout by the Hull red wipe test, the K and N ink test, and Vanceometer readings. No empirical test for smoothness and absorbency applies to all types of stocks. Print quality depends not only on the stock but on the ink and printing conditions. R.A.

Provisional Method for Testing the Fastness of Coloured Leathers to Organic Solvents
Society of Leather Trades Chemists

J. Soc. Leather Trades Chem., 42, 74–75 (March 1958)

The specimen of leather in contact with the specified uncoloured material (e.g. wool or cotton textiles) is agitated in the chosen solvent under standardised conditions. The change in colour of the specimen and the staining of the uncoloured material are assessed with the standard grey scales. J.W.D.

Provisional Method for Testing the Fastness to Rubbing of Light Leathers wetted from the Back with Organic Solvents
Society of Leather Trades Chemists

J. Soc. Leather Trades Chem., 42, 75–77 (March 1958)

The sample of leather is wetted from the back by contact with a felt pad soaked in the chosen solvent or mixture of solvents (e.g. dry-cleaning mixtures). The surface which is to be tested is then rubbed with a revolving dry felt pad, and the number of revolutions of the pad required to produce certain changes in the appearance of the finish is determined. The changes are assessed with the standard grey scale and are also described in terms of their character. J.W.D.

Determination of Apparent Second-order Transition Temperatures of Polymers
P. W. Jensen

J. Polymer Sci., 28, 635–638 (April 1958)

An apparatus is described whereby measurement of thermal elongation by strain gauges can be made at different temperatures. Elongation is plotted against temperature, and the intersection of the straight lines drawn through points on either side of an inflection point gives the apparent second-order transition temperature. A value of 79°c. is given for polyethylene terephthalate. W.R.M.

PATENTS

Measuring Relative Humidity
British Scientific Instrument Assocn. *BP* 795,031

Apparatus based on use of a porous ceramic which when dry has a specific resistance of 10^{6}–10^{13} ohm cm., e.g. slabs formed by sintering powdered TiO_2 at 900–1100°c. and then heating the slabs for some hours at 400–900°c. in a reducing atmosphere until a composition between TiO_2 and $TiO_{1.97}$ is attained. C.O.C.

Colour Contrast Lenses for Comparing Colours in Printing
C. Hirsch *USP* 2,792,744

Spectacles in which the lenses are the complement of the particular colour that is being viewed. When the colour printing is viewed through them the colour of the print is neutralised so that the printed matter stands out from the background and any defects can be immediately noticed. It also reveals whether or not the colour of ink being used is or is not an exact match to the sample pattern. C.O.C.

Apparatus for Testing the Wear Resistance of Yarns
"Licencia" Talalmanyokat Ertekesito Vallalat *USP* 2,788,655

Device for Selecting Dyes
S *BP* 796,256

A device which provides quick information about the properties of any given dye and indicates at a glance which dyes are suitable in combination comprises a page giving a number of examples and several masks which can be placed over it. These masks are perforated to reveal those dyes on the page which possess certain properties, each mask being perforated to show the same property of each dye. The masks are used singly or in combination. C.O.C.

Light Absorption of Dyes in Solution (IV p. 596)
Isolation of 2-Nitrophenol from a mixture of Mononitrophenols (IV p. 597)
Electron Microscope in the Surface-coating Industry (V p. 606)
Molecular Weight of Flax Pectin (VI p. 607)
Determination and Significance of Crystallite Size in Regenerated Cellulose Fibres (VI p. 607)
Tryptophan Content of Wool (VI p. 607)
Determination of *N*-Terminal Amino Acids of Collagen by the 2:4-Dinitrofluorobenzene Method (XII p. 617)

XV—MISCELLANEOUS

Art of Costume
A. M. Buck

J.S.D.C., 74, 329–330 (May 1958)

A summary of a well illustrated account of the trends of European fashion in clothes during the past few centuries. C.J.W.H.

Factor of Colour in Plastic Surgery
I. A. McGregor

J. Oil & Col. Chem. Assocn., 41, 288–290 (April 1958)

Skin colour is the resultant of three factors—blood supply, skin thickness, and skin pigmentation. Grafted skin does not always take on the colour of the normal skin in the vicinity of the graft, and adjustment may be made by tattooing pigments into the skin. For this purpose pigments must be non-toxic, chemically stable, and insoluble; this restricts the available range severely. Titania, chromium oxide, and yellow and red ferric oxides are used. J.W.D.

Technical Literature and its Assimilation
S. Burgess

J.S.D.C., 74, 458–463 (June 1958)

The problem of keeping abreast of the increasing volume of technical literature is discussed, special reference being made to the technique of fast reading, and the quality of scientific writing. AUTHOR

PATENT

Improving the Colour of Meat, etc.
Swift & Co. *USP* 2,798,812

A mixture of ascorbic acid and an antibiotic, e.g. aureomycin, prolongs the bright red colour life of meat or blood much beyond the period possible with ascorbic acid alone. C.O.C.

FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page viii*

Tuesday, 13th January 1959

Scottish Section. *Dyeing Unions Containing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Friday, 16th January 1959

Manchester Section. *The Degradation of Chemically Modified Celluloses by Alkali.* Dr. W. M. Corbett (British Rayon Research Association). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Monday, 19th January 1959

Huddersfield Section. Title to be announced later. Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I., F.S.D.C. (Joint meeting with the Huddersfield Textile Society.) Large Hall, Technical College, **Huddersfield.** 7.30 p.m.

Wednesday, 21st January 1959

Midlands Section. *The Solubility of Wool Dyes . . . Assessment and Practical Significance.* W. Beal, Esq., B.Sc. College of Technology, **Leicester.** 7 p.m.

Friday, 23rd January 1959

London Section. Annual Dinner and Dance. Waldorf Hotel, **London, W.C.2.** 7 p.m. for 7.30 p.m.

Thursday, 5th February 1959

Midlands Section. *Developments in Modern Dry-cleaning Techniques.* E. J. Davies, Esq., M.Sc. Gas Board Theatre, **Nottingham.** 7 p.m.

Friday, 6th February 1959

London Section. *Colour Photography.* Dr. R. W. G. Hunt (Kodak Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 10th February 1959

Northern Ireland Section. *Emulsion Thickenings—Possibilities in Textile Printing.* T. L. Dawson, Esq., B.Sc., Ph.D., A.R.I.C. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Scottish Section. *The Use of Antistatic Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). (Joint meeting with the Textile Institute.) St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Tuesday, 17th February 1959

Huddersfield Section. *The Use of Antistatic Agents in Textile Processing—Advantages and Disadvantages.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.) Silvios Cafe, **Huddersfield.** 7.30 p.m.

Friday, 20th February 1959

Manchester Section. *Emulsion Thickenings. Possibilities in Textile Printing.* T. L. Dawson, Esq., B.Sc., Ph.D., A.R.I.C., F.C.S. (Imperial Chemical Industries Ltd.). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Friday, 6th March 1959

London Section. *The Use of Antistatic Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

Midlands Section. Section Annual Dinner. George Hotel, **Nottingham.**

Tuesday, 10th March 1959

Northern Ireland Section. *Stenter Drying.* T. A. Uthwatt, Esq., B.A., A.M.I.E.E., and J. S. Woollatt, Esq., B.Sc. (Mather & Platt Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Scottish Section. Annual General Meeting. 7 p.m. Followed by *Modern Cloth Dyeing Principles and Machines.* K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). St. Enoch Hotel, **Glasgow.** 7·15 p.m.

Thursday, 12th March 1959

Midlands Section. *Dyeing and the Future.* J. Boulton, Esq., M.Sc.Tech., F.R.I.C., F.T.I., F.S.D.C., President of the Society. (Joint meeting with the Coventry Textile Society.) Courtauld's Acetate and Synthetic Fibres Laboratory, Lockhurst Lane, **Coventry.** 7 p.m.

Tuesday, 17th March 1959

Huddersfield Section. *Fast Shades on Wool-Cellulose Unions. Recent Advances in this Field.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Silvios Cafe, **Huddersfield.** 7.30 p.m.

Wednesday, 18th March 1959

Midlands Section. *Photoelectric Colorimeters . . . their uses and limitations in the Dyehouse.* J. V. Alderson, Esq., B.Sc., A.R.C.S. (Joint meeting with the British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.

Friday, 20th March 1959

Manchester Section. *Symposium on Re-active Dyes.* College of Science and Technology, **Manchester.** Further details later.

Thursday, 2nd April 1959

Midlands Section. *Colour Physics and Match Prediction.* (Lecturer to be announced later.) (Joint meeting with the Textile Institute.) Carpet Trades Ltd. Canteen, **Kidderminster.** 7 p.m.

Wednesday, 15th April 1959

Midlands Section. Annual General Meeting of the Section followed by a showing of New Scientific Films. King's Head Hotel, **Loughborough.** 7 p.m.

Friday, 17th April 1959

Manchester Section. Annual General Meeting. *Stenter Drying.* A. T. Uthwatt, Esq. (Mather & Platt Ltd., Research Dept.). The Textile Institute, **Manchester.** 7 p.m.

Tuesday, 21st April 1959

Huddersfield Section. Annual General Meeting. Followed by a Colour Film on *How the World Looks to a Colour-defective.* Cmdr. Dean Farnworth (United States Naval Research). Silvios Cafe, **Huddersfield.** 7.30 p.m.

Friday, 24th April 1959

London Section. Annual General Meeting. Commander Dean Farnworth (United States Navy) will show and discuss his film *How the World Looks to a Colour-defective.* Waldorf Hotel, **London W.C.2.** 6 p.m.

ADDRESSES WANTED

Buckley, H. H., *formerly of* G.P.O. Box 3695, Sydney, Australia

Cameron, M. C., *formerly of* 11 Thirlmere Gardens, Belfast, N. Ireland

Ciprut, J. V., *formerly of* Technical College, Dyeing Dept., Bradford, Yorkshire

Drzewicki, M., *formerly of* Flat 2, 11 Rectory Road, West Bridgford, Nottinghamshire

Gale, R. A., *formerly of* 32 Wordsworth Street, Hapton, near Burnley, Lancashire

Harris, A., *formerly of* "Laurel Bank", 36 High Street, Newton-le-Willows, Lancashire

Harvey, H. C., *formerly of* 77 Edinburgh Road, Congleton, Cheshire

Heaton, R., *formerly of* 142 Browning Road, Manor Park, London E.12

Hollis, D. J., *formerly of* "Kinder Spur", Hayfield, Stockport, Cheshire

Hubler, B., *formerly of* Park Hotel, Oak Avenue, Manningham, Bradford 8

Jackson, J. B., *formerly of* 36 Arden Road, Crumpsall, Manchester 8

Jliadis, S., *formerly of* Nottingham and District Technical College, Shakespeare Street, Nottingham

Kabil, R., *formerly of* Sefitex and Company, Mataria Street, Zeitoun, Cairo, Egypt

Kothawala, Amin A., *formerly of* 36 Briarwood Drive, Wibsey, Bradford 6

Kubba, S., *formerly of* 10 Malone Road, Belfast, N. Ireland

Le Bek, I. A. G., *formerly of* 8 Lad-Hill Lane, Greenfield, near Oldham, Lancashire

Maschas, L., *formerly of* 43 St. John's Road, Huddersfield

Midgley, F. B., *formerly of* "Clova", 57 Jeremy Lane, Heckmondwike, Yorkshire

Oxley, R., *formerly of* 27 Hayling Road, Sale, Manchester

Ramadan, A. S., *formerly* c/o Beida Dyers, Dyeing Department, Alexandria, Egypt

Rahman, S. M. K., *formerly of* Lock View Hall, 1025 Gt. Western Road, Glasgow

Richardson, D., *formerly of* 139 Laund Road, Salendine Nook, Huddersfield

Slater, W. K., *formerly of* 85 Hope Road, Sale, Cheshire

Smith, L., *formerly of* 80 Fair View Road, Bacup, Lancashire

Troxler, M. D., *formerly of* Georgia Institute of Technology, Box 680, Ga. Tech., Atlanta, U.S.A.

Tsien, P. C., *formerly of* 1332 Tenan Road, Shanghai, China

Wahba, F. S., *formerly* c/o Beida Dyers S.A.E., P.O. Bag., Alexandria, Egypt

Wailes, N. T., *formerly of* "Hamewith", 4 Kingsway Terrace, Dundee, Scotland

Walker, T. H., *formerly of* J. L. Stifel & Sons Inc., 339 Main Street, Wheeling, W. Va., U.S.A.

Zwicky, A. M., *formerly of* 23 West Leight Street, Crumpsall, Manchester 9

MEMBERS' CHANGES OF ADDRESS

Bergman, Z., *formerly of* 28 Headington Avenue, Coventry, *to* c/o O. Bergman, 3200 Swede Road, Midland, Mich., U.S.A.

Budding, J., *formerly of* Woodsley Hall, Clarenden Road, Leeds 2, *to* c/o The Dept. of Colour Chemistry and Dyeing, The University, Leeds 2.

Burdett, B. C., *formerly of* Woodsley Hall, Clarendon Road, Leeds 2, *to* The Dept. of Colour Chemistry and Dyeing, The University, Leeds 2

Chingros, Chris., *formerly of* White Street, Lunenburgh, Mass., U.S.A., *to* 105 Cedar Street, Fitchburg, Mass., U.S.A.

Clayton, F. H., *formerly of* Wernferig, Goytre, near Pontypool, *to* Greythwaite, Butley Park, Prestbury, Cheshire

Clayton, J., *formerly of* 16 Prospect Avenue, Windhill, Shipley, *to* 11 Kitson Street, Windhill, Shipley

Collins, I. R., *formerly of* 11 Blenheim Mount, Manningham, Bradford 8, *to* Fabrica de Tejidos "La Union" Ltd., Casilla 2066, Lima, Peru, South America

Courtney Harwood, F., *formerly of* Roefield, Felden Lane, Boxmoor, Herts., *to* 6 Fernpark Close, Exeter, Devon

Grant, A. C., *formerly of* Imperial Chemical Industries Ltd., Imperial House, Donegal Square East, Belfast, N. Ireland, *to* Imperial Chemical Industries, Dyestuffs Dept., 4 Blythswood Square, Glasgow C.2

Hall, Wm. M., *formerly of* 13 Kirkliston Park, Belfast, N. Ireland, *to* 369 Beersbridge Road, Belfast, N. Ireland

Heggie, James, *formerly of* Denburn Dyeworks, Kirkcaldy, *to* 108 Nicol Street, Kirkcaldy

Holt, J., *formerly of* 27 Milton Drive, Boreham Wood, Herts., *to* 70 Hillside Gardens, Barnet, Herts.

Jabr, S. U., *formerly of* P.O. Box 369, Beirut, Lebanon, *to* P.O. Box 3072, Beirut, Lebanon

Jordanides, T. A., *formerly of* P.O. Box 8193, Charlotte 8, N.C., U.S.A., *to* Aristodimou 4, Athens, Greece

Lee, K. R., *formerly of* I.C.I. Ltd., 7 Donegal Square East, Belfast, *to* Imperial Chemical Industries Ltd., Sales Centre (Industrial Products), Billingham, Co. Durham

Lock, L. C., *formerly of* St. Thomas Rectory, McLaren Street, North Sydney, N.S.W., *to* 143 Lambton Road, Charlestown, N.S.W., Australia

McInnes, A. A., *formerly of* 64 Grangefield Crescent, Castlereagh Road, Belfast, N.I., *to* 29 Abbeydale Parade, Crumlin Road, Belfast, N.I.

Makepeace, T. H., *formerly of* West Port House, 15 Kerr Street, Portrush, Co. Antrim, Northern Ireland, *to* New Somerset House, Coleraine, County Londonderry, Northern Ireland

Masheter, J. J., *formerly of* Covendale, Oxford Road, Macclesfield, Cheshire, *to* 76 Bradford Street, Bocking, Braintree, Essex

Robinson, A., *formerly of* 421 Leyland Lane, Leyland, near Preston, *to* Holborn Stores and Post Office, 20 Holborn Hill, Aughton, Ormskirk, Lancs.

Stevenson, R. F., *formerly of* Blackbrook House, Blackbrook, Duffield, near Derby, *to* Castle Hill House, Vicarage Lane, Duffield, near Derby

Sumner, N. L., *formerly of* 7 Park Drive, Westville, Natal, South Africa, *to* 17 Woodlands Avenue, Westville, Natal, South Africa

Sylvester, C. A., *formerly of* E. I. Du Pont de Nemours & Company, Inc., Technical Lab., P.O. Box 386, Wilmington 99, Delaware, U.S.A., *to* E. I. Du Pont de Nemours & Company, Inc., Trade Relations Section, Organic Chemicals Department, Room 7430, Nemours Building, Wilmington 98, Delaware, U.S.A

APPOINTMENTS etc

Advertisements relating to Appointments Vacant, Appointments Wanted, and Miscellaneous Items are invited for insertion on this page. Advertisements of Appointments Wanted are gratis to members, but must not exceed twenty-four words.

All inquiries relating to Advertisements should be addressed to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

Replies may be addressed Box —, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire, where all communications relating to these Advertisements are treated in strict confidence.

APPOINTMENTS VACANT

A Printing Colourist is required by Rainshore Bleaching and Dyeing Co. Ltd., Norden, Rochdale. This is a new position and is a progressive appointment for a suitable applicant. Please write in the first instance, stating qualifications, experience and approximate salary requirements. All applicants will be interviewed.

DYEING AND FINISHING

BRITISH NYLON SPINNERS LIMITED invite applications for a vacancy caused by the completion of their new Experimental Dyehouse. The work will be concerned primarily with the dyeing and finishing of nylon woven fabrics. Candidates must have had experience in the handling and processing of fine fabrics of man-made fibres, and should, for preference, have been associated with experimental and development work rather than normal production. A degree in textile chemistry or textiles is a very desirable qualification, but a lower academic level might be acceptable if combined with a particularly wide and relevant practical experience. The ability to co-operate with people of widely differing backgrounds, working in similar fields, is considered of the first importance. Candidates should be 27 to 37 years of age. A good starting salary will be paid, there will be opportunities for progression on merit and conditions of employment are of the highest. Rented houses may be available to married staff coming from outside this area. Applications should be addressed to the Personnel Manager, Pontypool, Mon., to reach him by September 30th.

TEXTILE RESEARCH AND DEVELOPMENT

THE Research & Technical Service Department of a Company in Lancashire requires a Chemist or Textile Technologist, preferably not over 30, with a degree or an equivalent qualification. The work is concerned with the development and production of a wide range of fabrics for industrial uses. Knowledge of textile processes, particularly in relation to synthetic fibres, would be an advantage, though not essential.

The successful candidate would ultimately take over responsibility for one section of the laboratories.

The Company has a five-day week, and operates a Pension and Life Assurance Scheme. Replies to Box V153

LABORATORY VACANCIES
DOMESTOS LTD.

YOUNG MALE GRADUATES, or equivalent, preferably with degree in chemistry as RESEARCH ASSISTANTS. These opportunities are as a result of the Company's increasing range of products in the detergent and allied fields. Applications in confidence to—Personnel Manager, Domestos Limited, College Works, Albion Row, Newcastle-upon-Tyne 6.

APPOINTMENTS WANTED

CHEMIST/TECHNOLOGIST, B.Sc. (Hons.Col.Chem.). Age 49, 27 years' experience high standard Bleaching, Dyeing, Finishing, Cotton, Wool, Silk, Screen Printing, Dyehouse Management, seeks position. Box W601

NEW MEMBERS

Persons desirous of joining the Society as Ordinary Members or Junior Members can obtain Application Forms from the General Secretary, or from the Honorary Secretary of any Section of the Society

Bennett, W. E., The Hexoran Co. Ltd., Derwent Street, Belper
Bratt, A. E., The Clayton Dyestuffs Co. Ltd., Ashton New Road, Manchester 11
Buckner, J. M., The William Carter Co., Barnesville Georgia, U.S.A.
Cadman, F., Messrs. Joseph Clare Ltd., Frenches Dyeworks, Greenfield, Oldham, Lancs.
Cheatham, A. F. Jnr., 2811 Cascade Road, S/W, Atlanta, Georgia, U.S.A.
Corbett, E., 25 Recreation Road, Longford, Coventry
Crawshaw, G. H., 17 Richmond Road, Leeds 6
Dossa, M. F., North Carolina State College of Agriculture and Engineering, Raleigh, U.S.A.
Hickman, W. S., 3 Clinton Place, West Derby, Liverpool
Hill, R., 52 Dale Street, Accrington, Lancs.
Howarth, C., Messrs. Barford Bros., North Street, Luton, Beds.
Jaeckel, S. M., 46 Woodhall Road, Wollaton Road, Nottingham
Joseph, A. A., Messrs. Jeanne Feathers Ltd., 10 Kendall Place, London W.1
Miller, Q. B., 9 Thornhill Road, Rowditch, Derby
Moretti, Giorgio, Tintorice Movetti, Cinisello Balsamo, Milano, Italy
Myers, M. R., 42 Waldeck Road, Sherwood, Nottingham
Olley, M. J., 14 Woodland Road, Claybrooke Magna, near Rugby
Swift, K. A., Allied Colloids (Bradford) Ltd., 18a North Parade, Bradford
Widdop, D., 6 Moorend View, Pellon, Halifax
Zollinger, H., 9 Im Marteli, Binningen, Switzerland

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